



Evaluation of Adhesive Bond Primers for Repair Bonding of Aluminum

2011 DoD Joint Committee on Tactical Shelters (JOCOTAS)

2 November 2011



Materials and Manufacturing Directorate

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Overview



- **Background**
- **Materials and Processes**
- **Tests and Test Methods**
- **Lap Shear and Peel Data**
- **Wedge Test Data**
- **Conclusions**
- **Future Work**



Background



- **Large Screening Effort to Evaluate Low-VOC Bond Primers**
- **Primary Focus is to Eliminate Chromated Bond Primers**
- **Working with Industry-Government Non-Cr Adhesive Bond Primer Team**
 - Effort is part of the larger picture but also has USAF focus
- **Desire Performance Equivalent to that Currently Obtained with Chromated Primers**
 - Cytec's BR 127 and BR 6747-1, primarily
- **Bond Strength and Moisture Durability are Both Important**
- **Looking for Primer that Works Well with Multiple Adhesives on Adherends Prepared Using Grit-Blast/Sol-Gel (GBSG)**
 - Currently, GBSG is used exclusively with Cytec's BR 6747-1 primer
 - Cytec's FM 73 adhesive is not recommended for use with GBSG
 - 3M's AF 163-2 adhesive tends to fail at its interface with BR 6747-1



Materials and Processes



- **Five Waterborne Adhesive Bond Primers**
 - Cytec's BR 6747-1 (zero-VOC, chromated)
 - Cytec's BR 6747-1NC (zero-VOC, nonchromated - no inhibitor)
 - Cytec's BR 6700-1 (low-VOC, nonchromated corrosion inhibitor)
 - 3M's EW 5000 (low-VOC, chromated)
 - 3M's EW 5000 AS (low-VOC, nonchromated corrosion inhibitor)
- **Spray Applied Using Conventional Air Gun**
 - Dry film thicknesses: 0.0025 mm - 0.0063 mm (0.0001 - 0.00025 in); measured using Fischer Isoscope MP30E-S with ETA3.3H probe (directly on PAA adherends and on travelers for GBSG adherends)
- **Cured in an Air-circulating Oven at 121°C (250°F) for 60 Mins after Air Dry at Ambient Conditions for 30 Mins**



Materials and Processes (cont.)



- **Seven Adhesives Evaluated with Each Primer**
 - Henkel's Hysol EA 9696 and Hysol EA 9628
 - Cytec's FM 73M, FM 209M, and FM 300-2M
 - 3M's AF 163-2M and AF 500M
- **Mat Carrier**
- **290g/m² (0.06 psf) Areal Weight**
- **Cured per Manufacturers' Recommendations Under Positive Pressure**
 - 121°C (250°F) for 60-90 Minutes



Materials and Processes (cont.)



- **2024-T3 (bare) Aluminum Adherends**
- **Two Surface Preparations**
 - Phosphoric Acid Anodize (PAA) per ASTM D 3933
 - First choice for aluminum prebond surface preparation
 - Used in USAF for rework and preparation of repair doublers
 - Grit-Blast/Sol-Gel (GBSG)
 - Al_2O_3 grit-blast followed by application of AC Tech AC-130 (Boegel-EPII) sol-gel solution; no rinse and ambient dried
 - Used in USAF for preparation of on-aircraft adherends



Tests and Test Methods

- **Tensile Lap Shear (ASTM D 1002)**
 - Ambient temperature at 22°C (72°F)
 - Hot/Wet at 82°C (180°F) after conditioning for 90 days at 60°C (140°F) and 95-100% relative humidity (RH)
- **Metal-to-Metal Climbing Drum Peel (ASTM D 1781)**
 - Ambient temperature at 22°C (72°F)
- **Wedge Test (ASTM D 3762)**
 - Conditioned at 60°C (140°F) and 95-100% RH for 28 days
 - Conditioned at 49°C (120°F) and 95-100% RH for 28 days*
- **Five Specimens per Test for Each Adhesive/Primer/Surface Preparation Combination**

* These data are not reported in the paper and will not be presented; will be in a comprehensive report



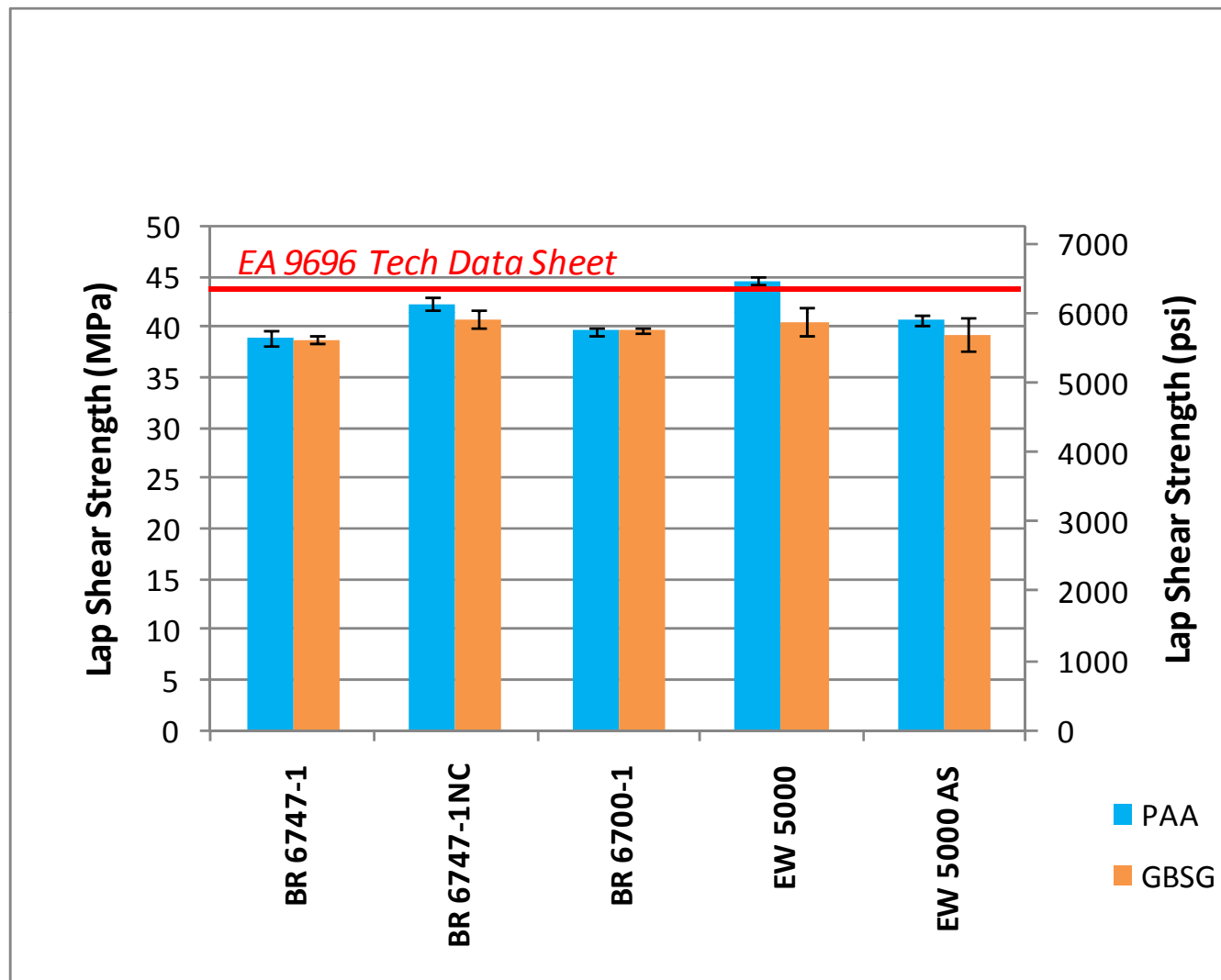
The Wedge Test

- **Used Extensively by AFRL for Screening to Assess Bonded Joint Moisture Durability**
- **Considered by AFRL to be a Semiquantitative Test**
 - Initial crack lengths provide important information (w.r.t. test severity)
 - Crack extensions can be useful, but are not the bottom line
 - Most consideration is given to failure modes after conditioning
 - Desire failure modes are 95% or greater cohesive within the adhesive
- **Caveats**
 - Useful for comparing surface preparations (including primers)
 - Many factors affect the test; must only vary the factor being evaluated
 - Several adhesive characteristics affect the test: modulus, bondline thickness, carrier, degree of cure, hot/wet properties
 - Test cannot be quantitatively correlated with in-service performance



EA 9696 Adhesive

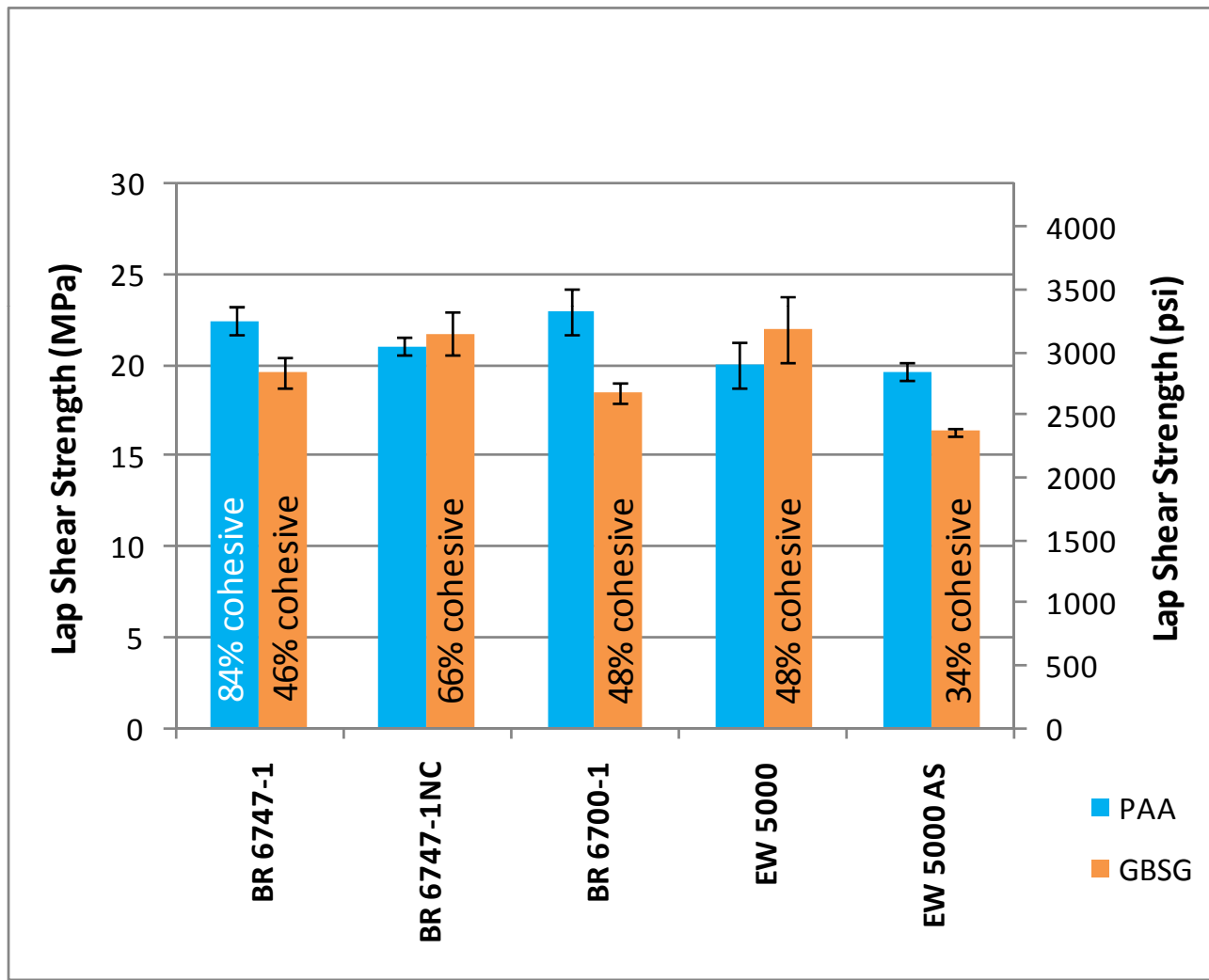
22°C (72°F) Lap Shear





EA 9696 Adhesive

82°C (180°F) Wet Lap Shear





EA 9696 Adhesive

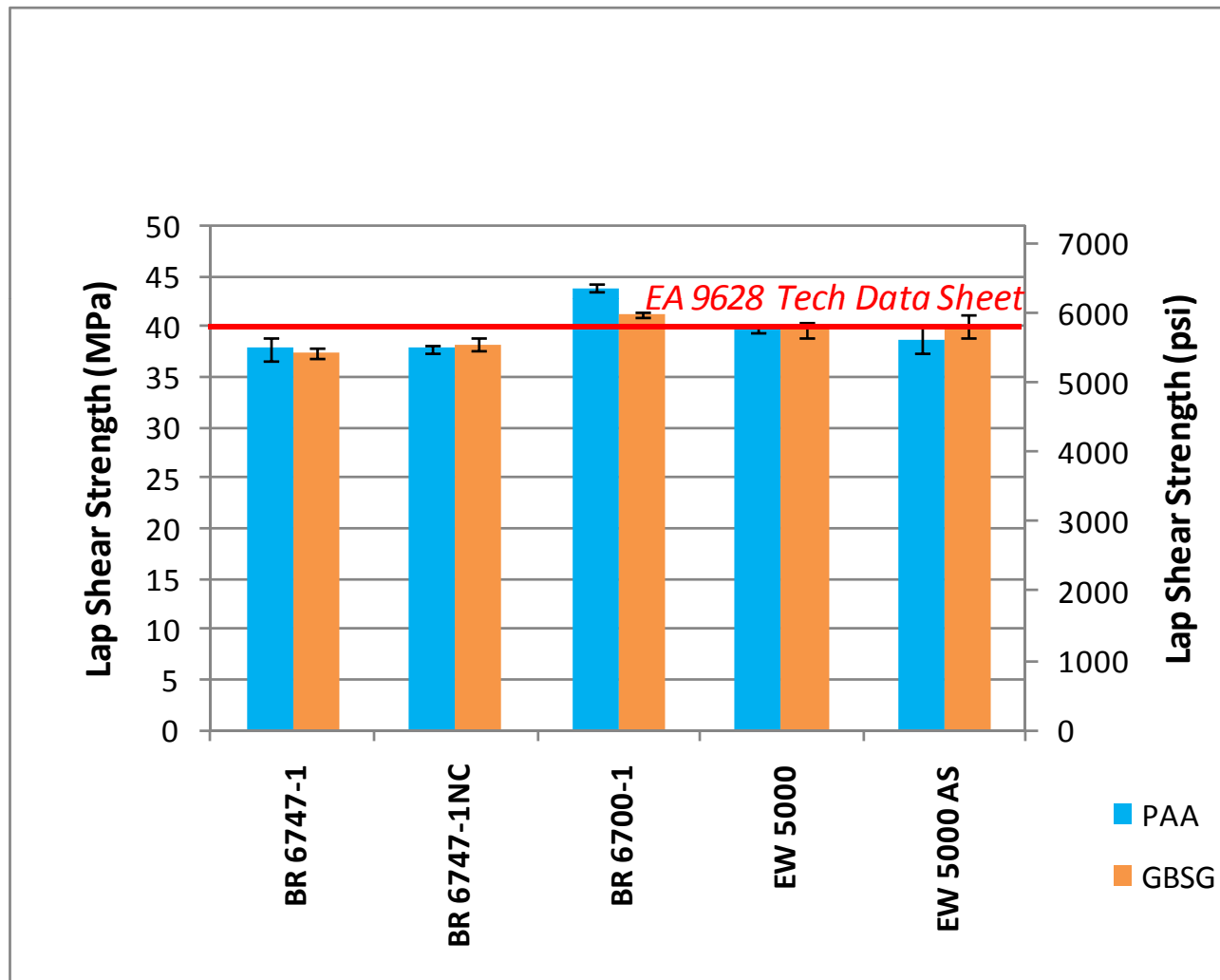
22°C (72°F) Climbing Drum Peel





EA 9628 Adhesive

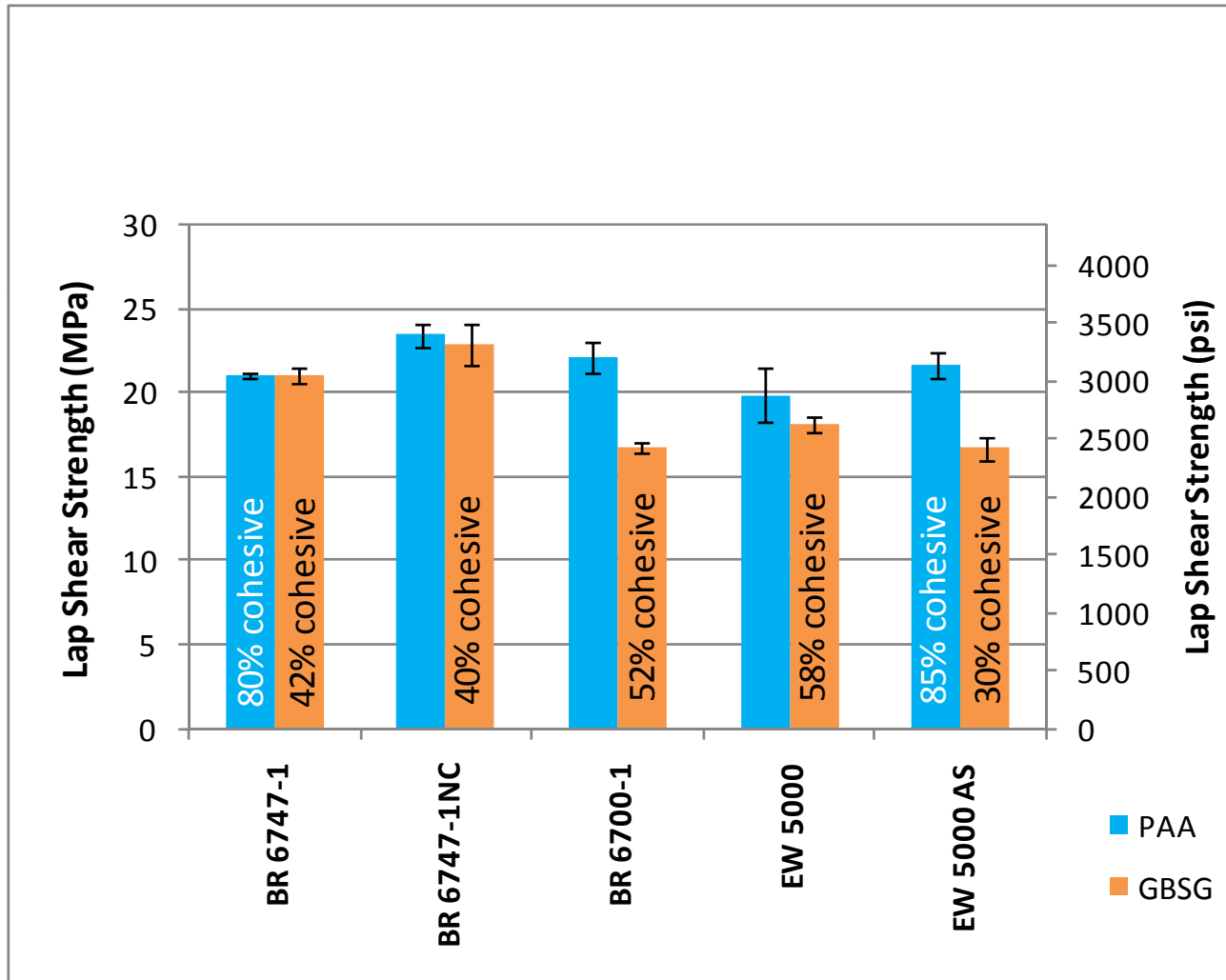
22°C (72°F) Lap Shear





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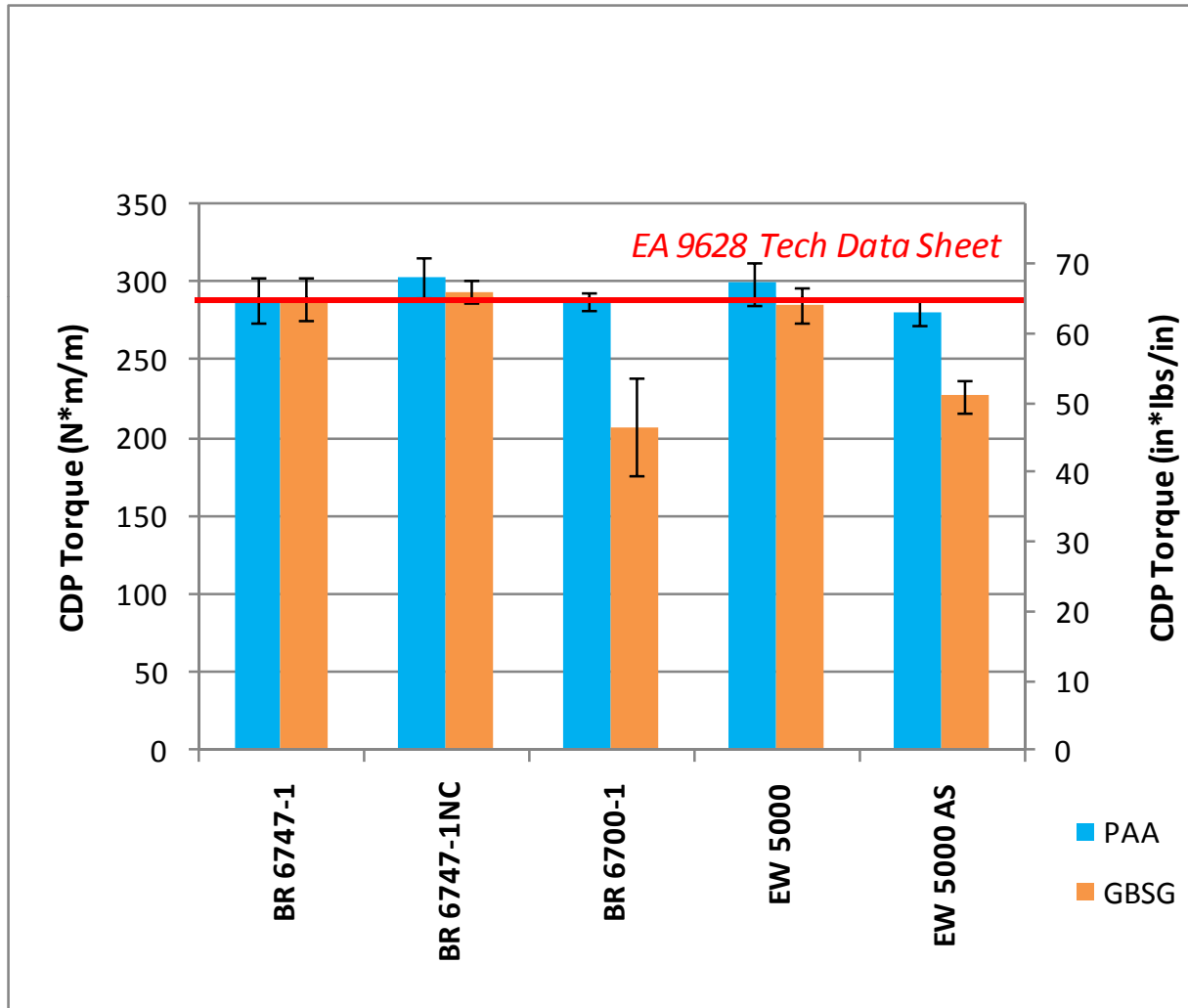
82°C (180°F) Wet Lap Shear





EA 9628 Adhesive

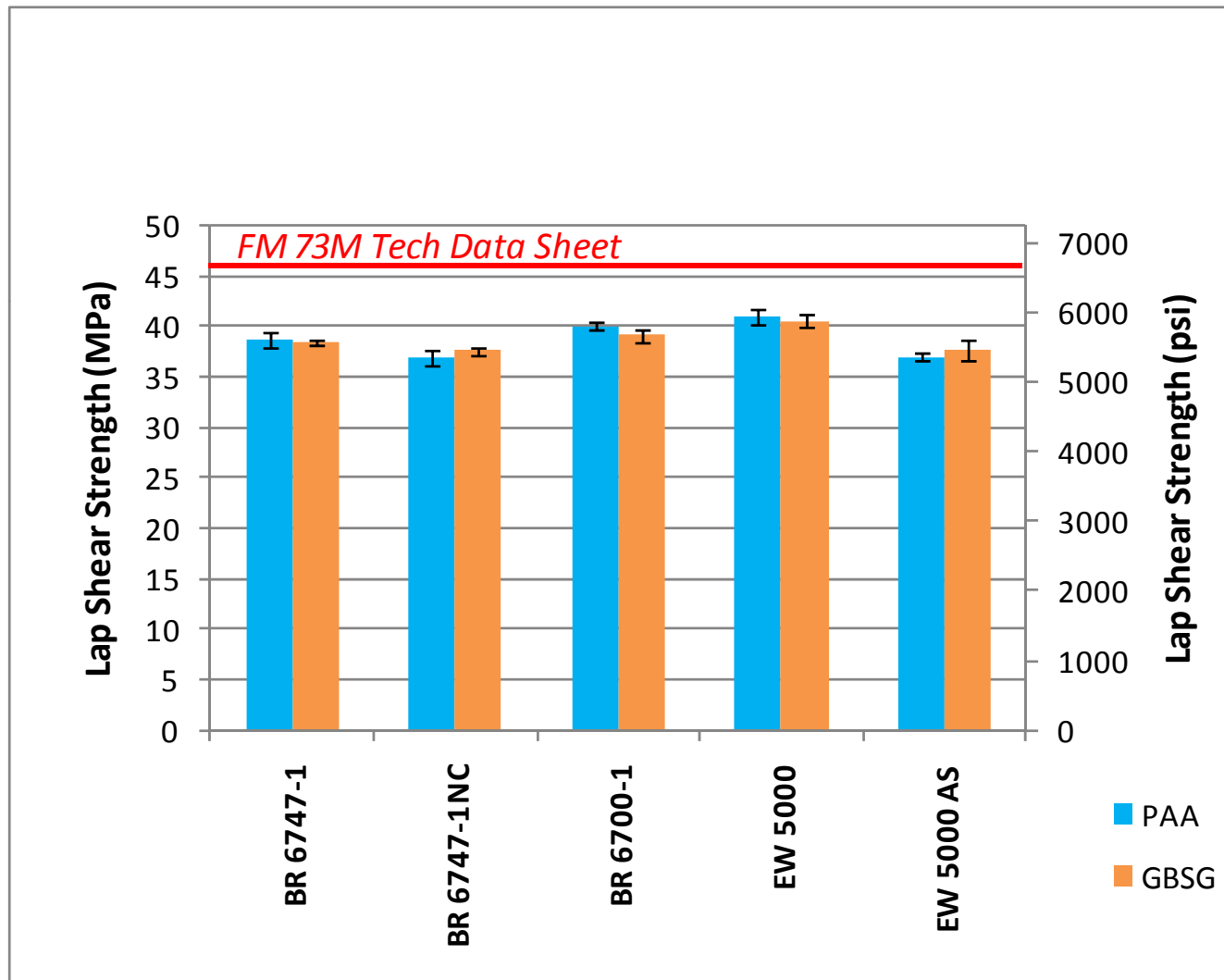
22°C (72°F) Climbing Drum Peel





FM 73M Adhesive

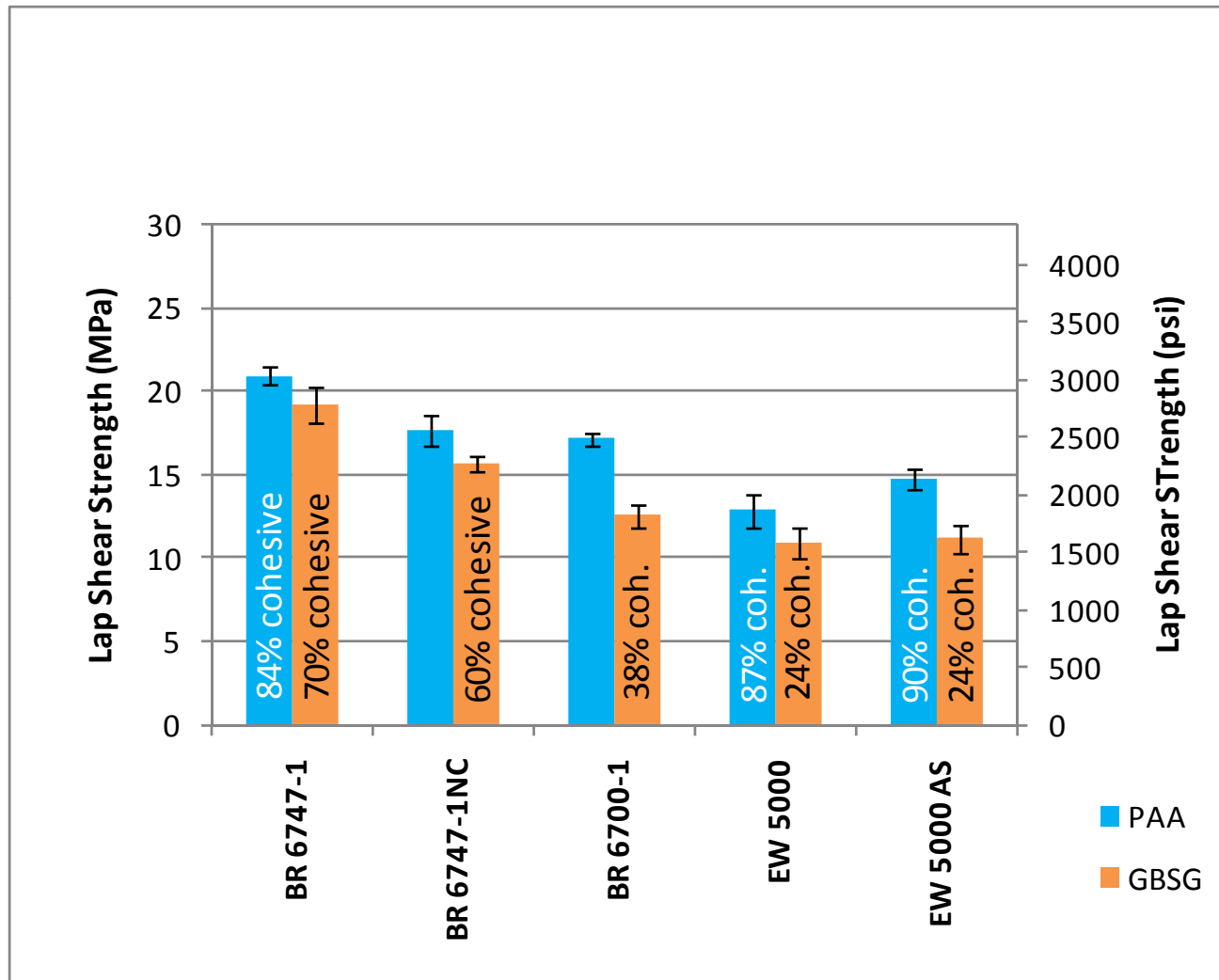
22°C (72°F) Lap Shear





FM 73M Adhesive

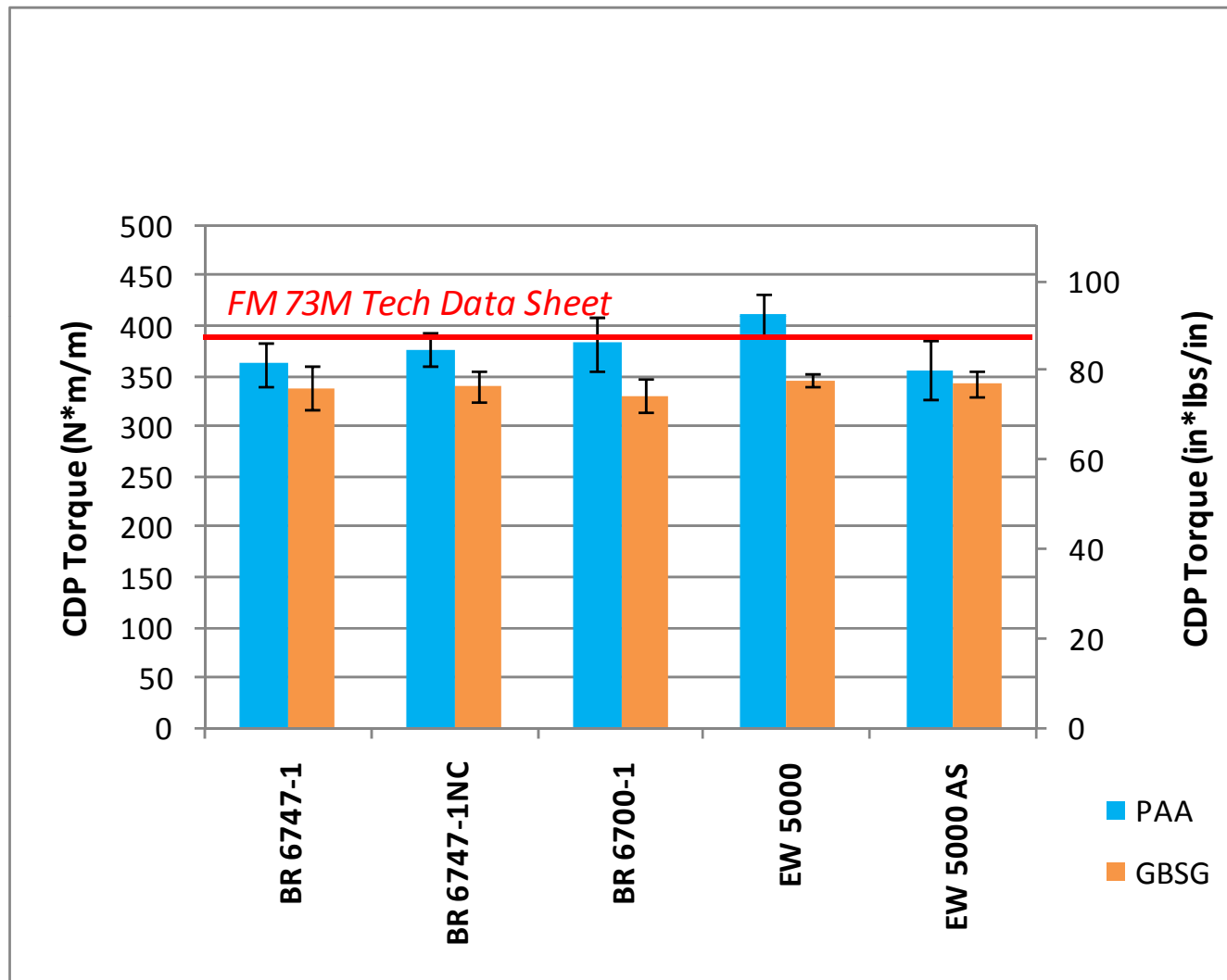
82°C (180°F) Wet Lap Shear





FM 73M Adhesive

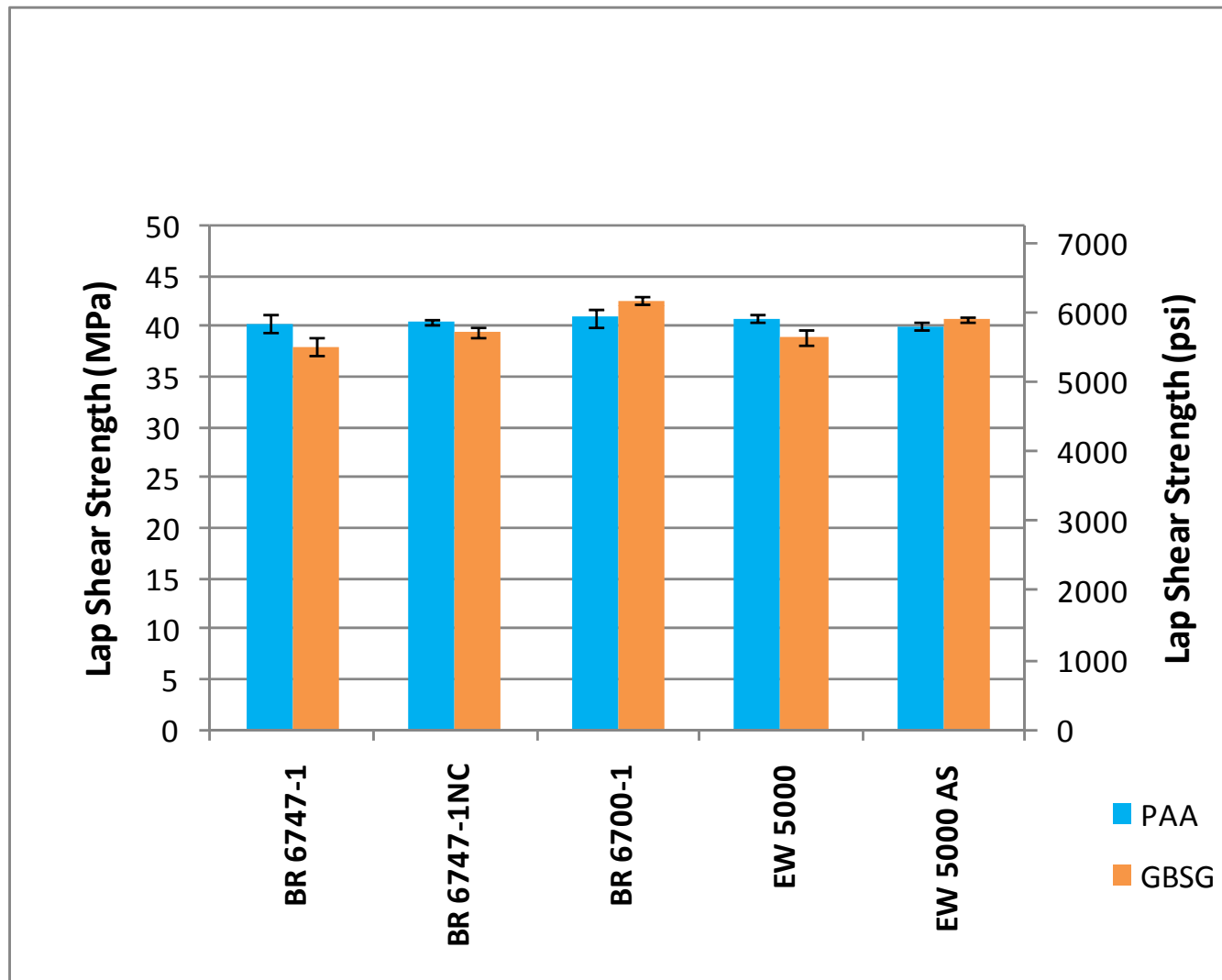
22°C (72°F) Climbing Drum Peel





FM 209M Adhesive

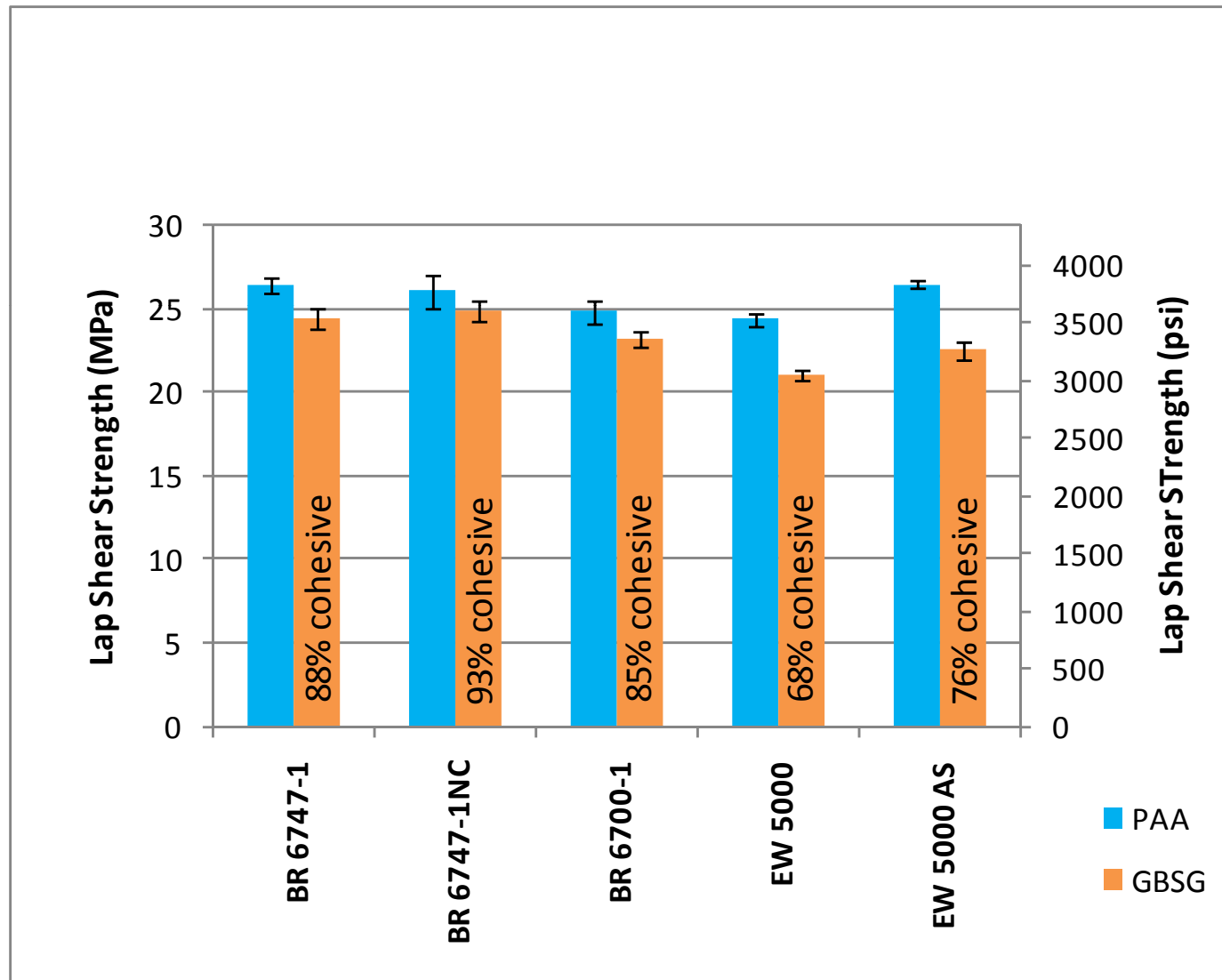
22°C (72°F) Lap Shear





FM 209M Adhesive

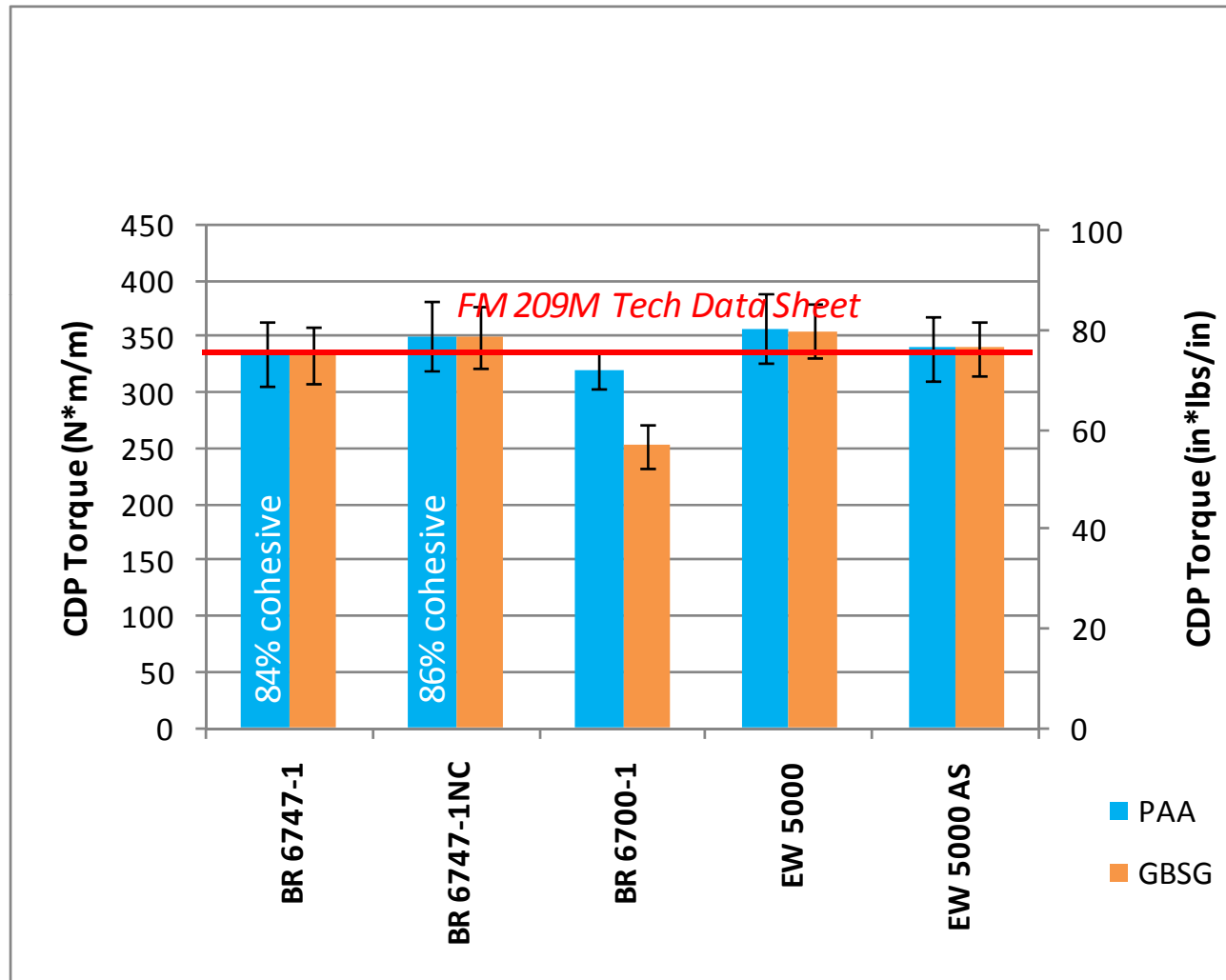
82°C (180°F) Wet Lap Shear





FM 209M Adhesive

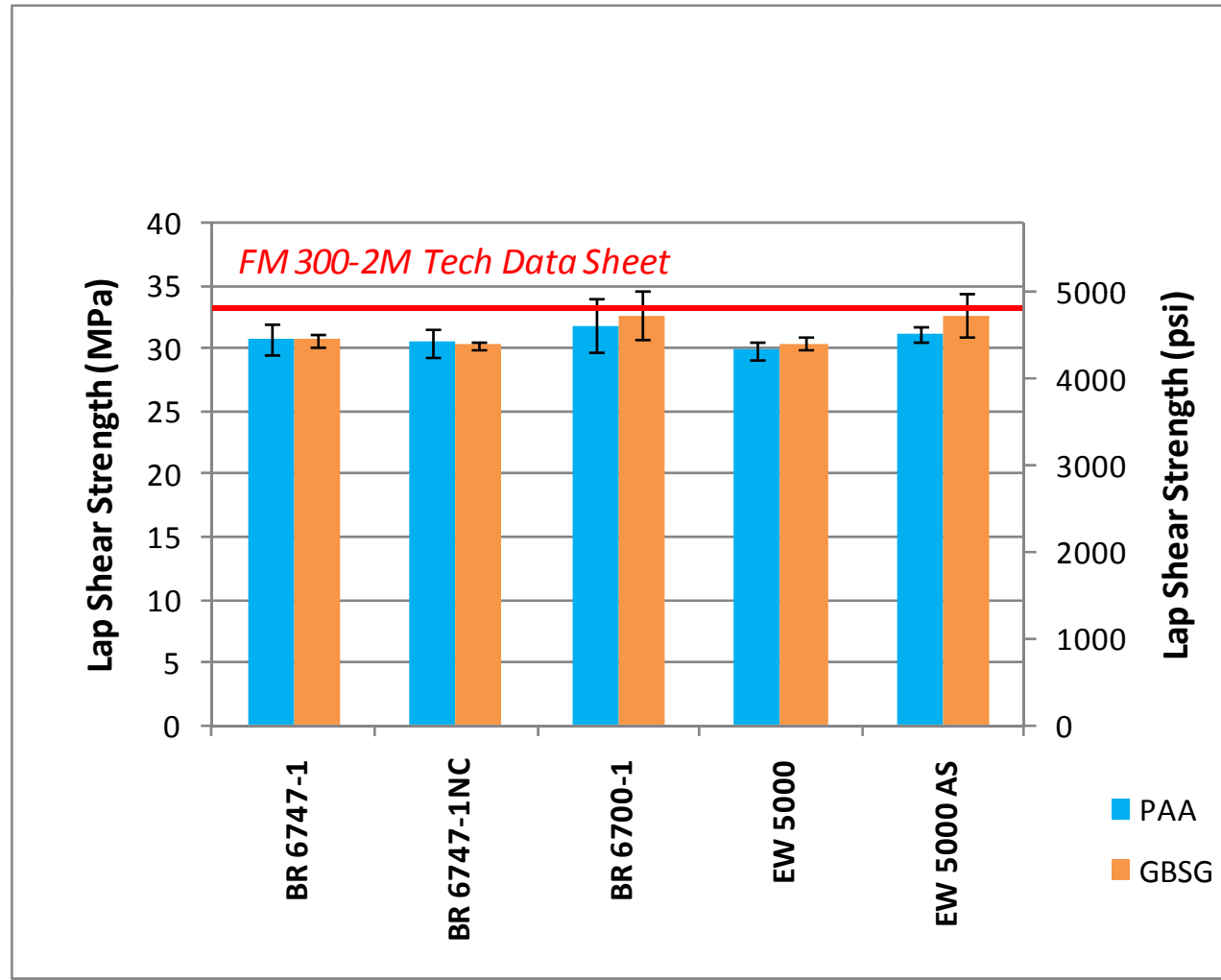
22°C (72°F) Climbing Drum Peel





FM 300-2M Adhesive

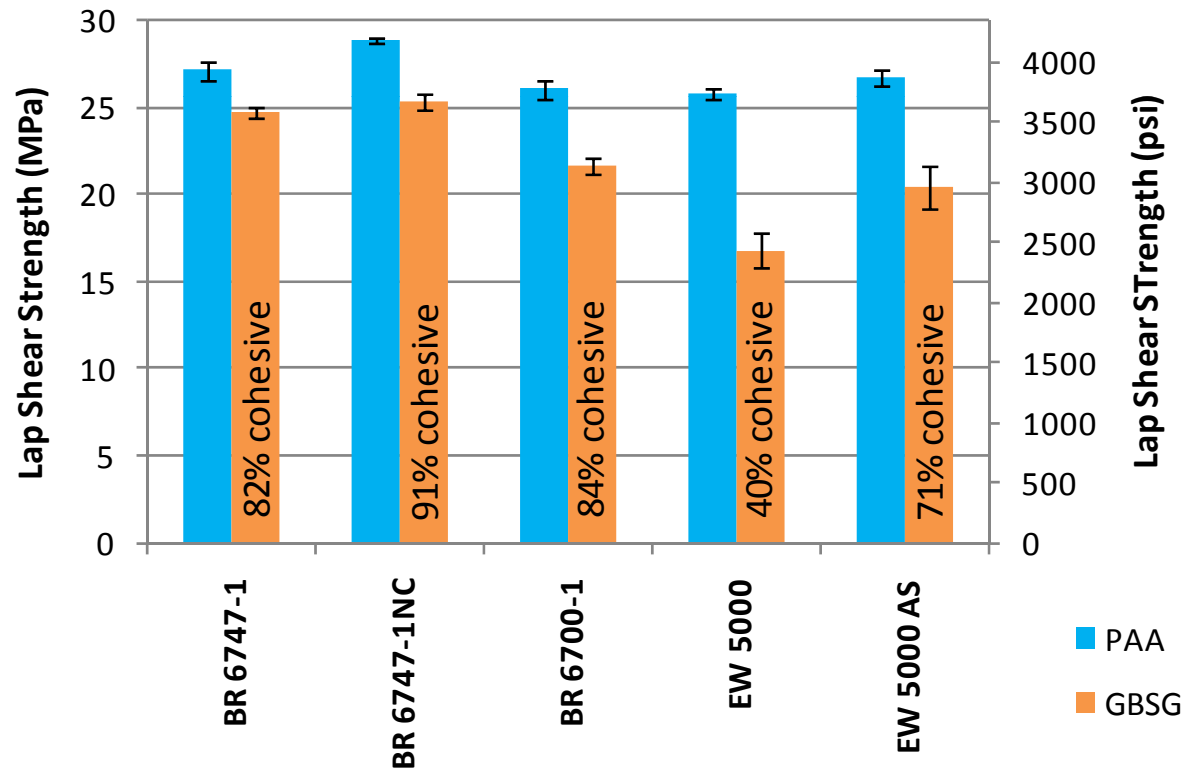
22°C (72°F) Lap Shear





FM 300-2M Adhesive

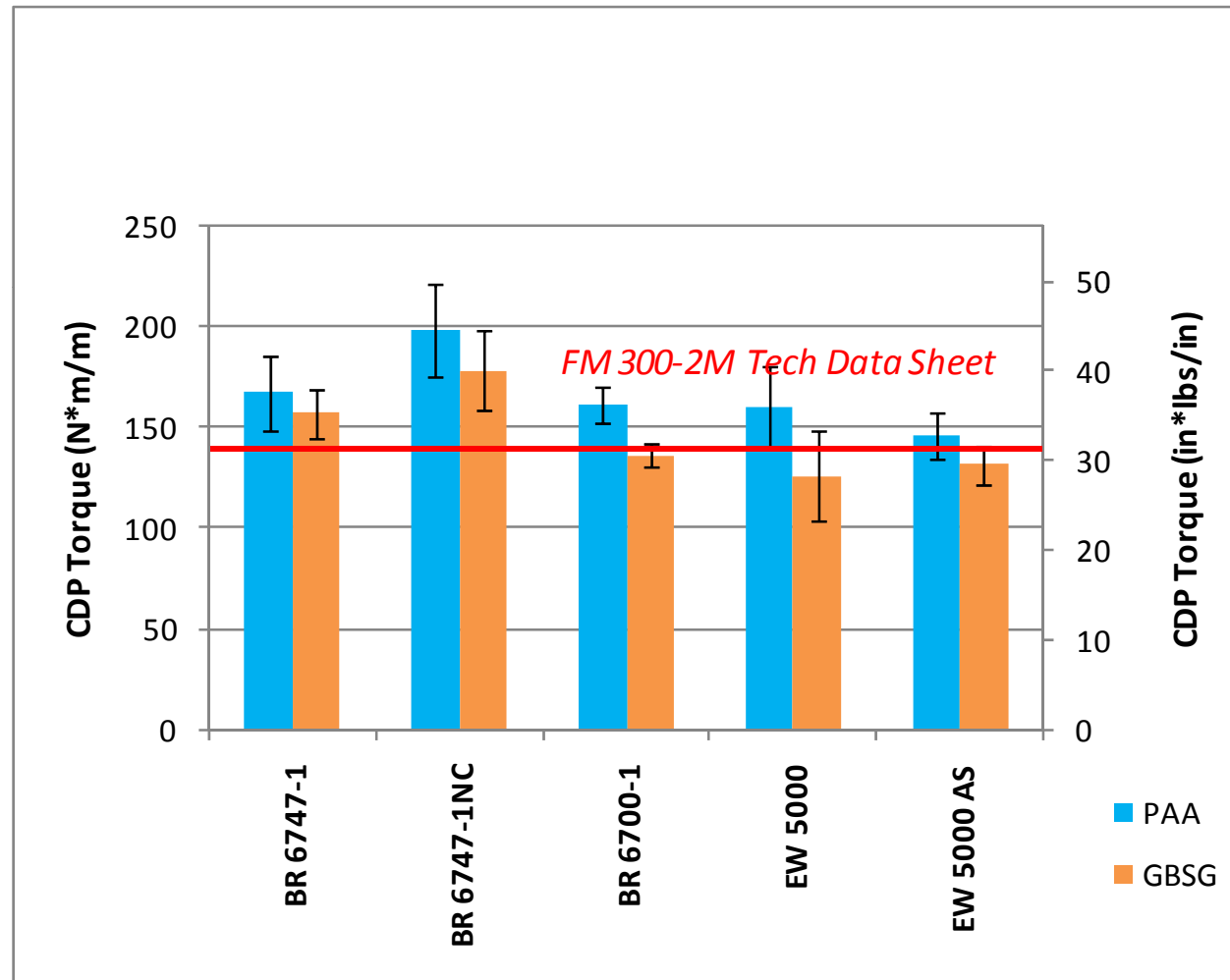
82°C (180°F) Wet Lap Shear





FM 300-2M Adhesive

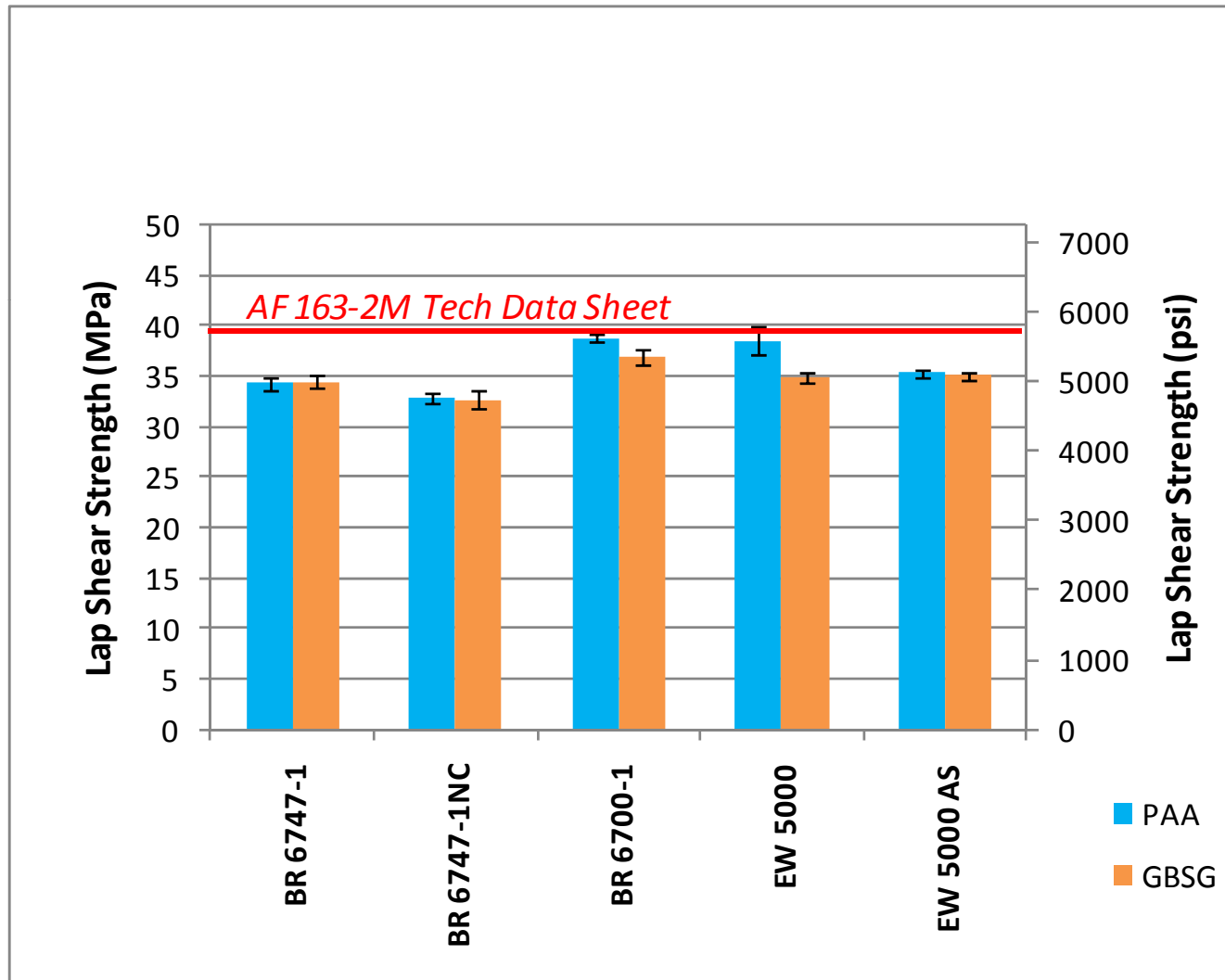
22°C (72°F) Climbing Drum Peel





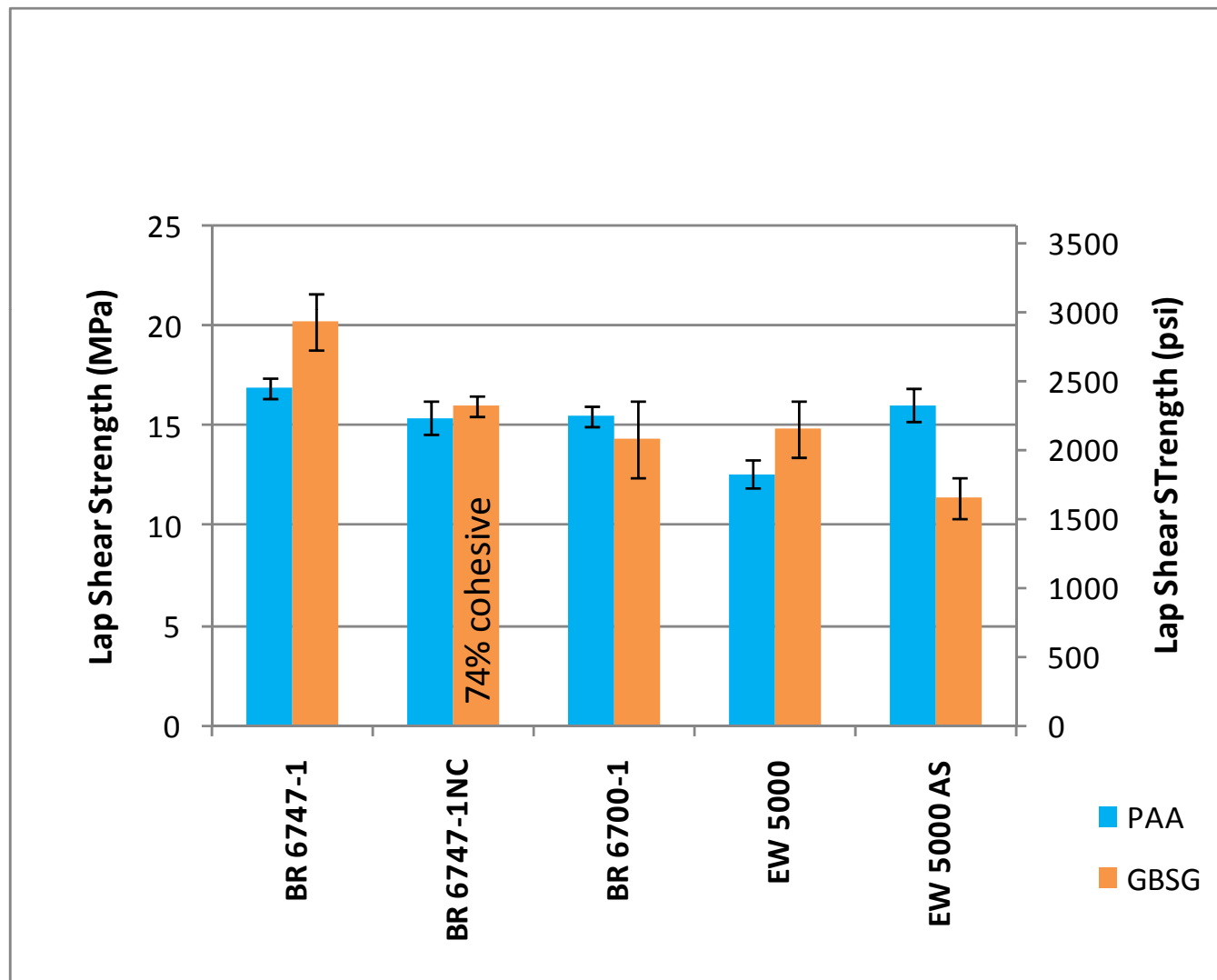
AF 163-2M Adhesive

22°C (72°F) Lap Shear





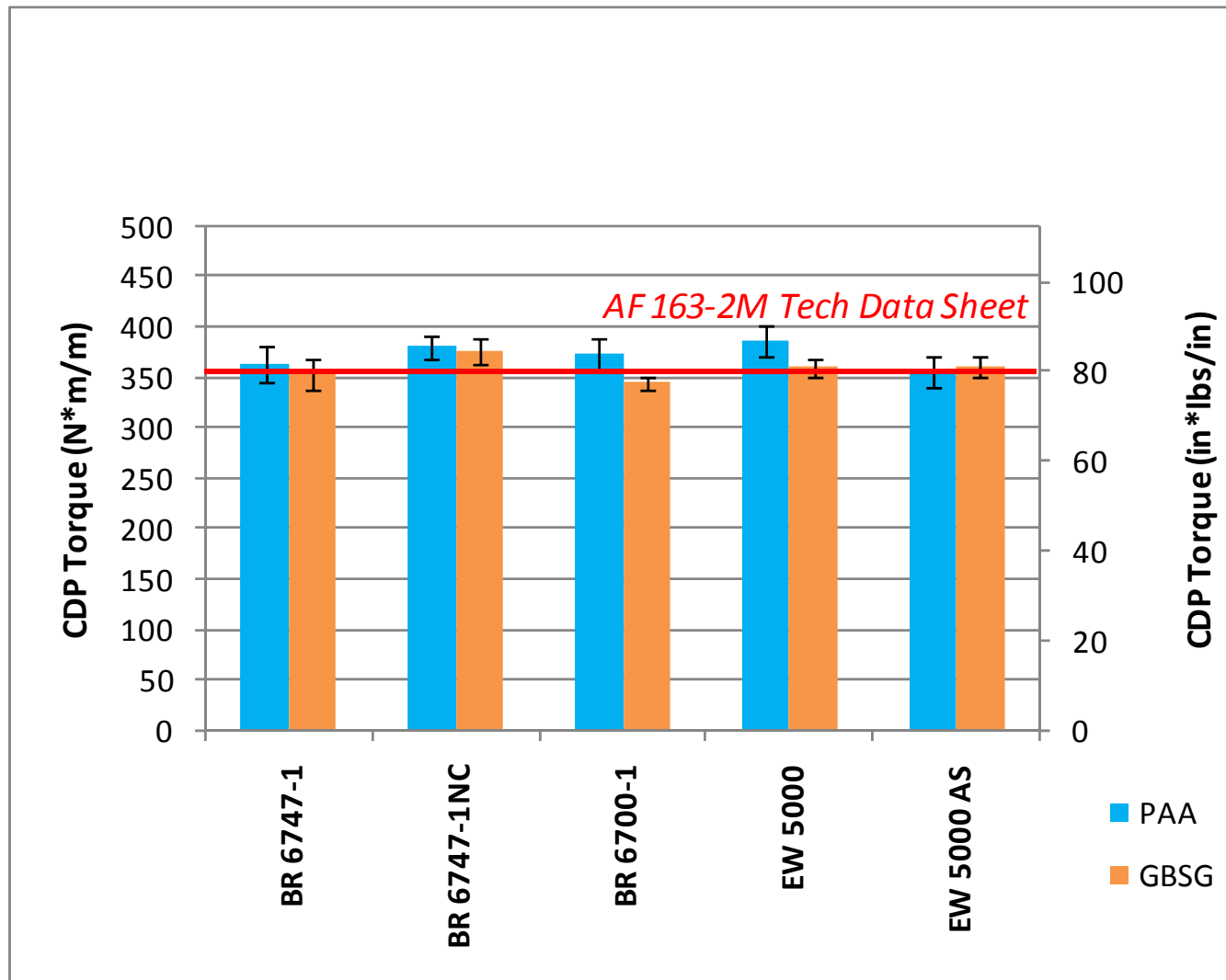
AF 163-2M Adhesive 82°C (180°F) Wet Lap Shear





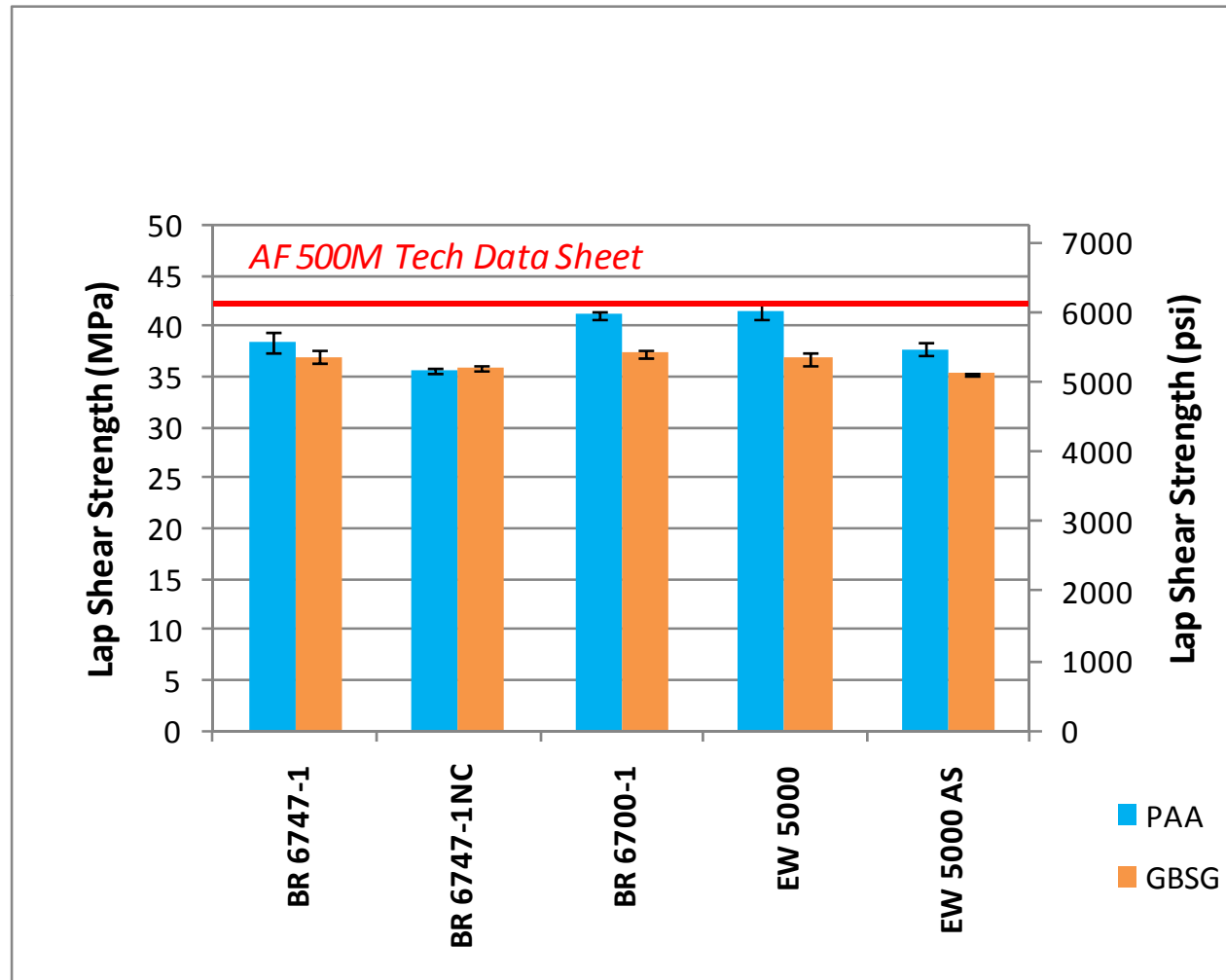
AF 163-2M Adhesive

22°C (72°F) Climbing Drum Peel





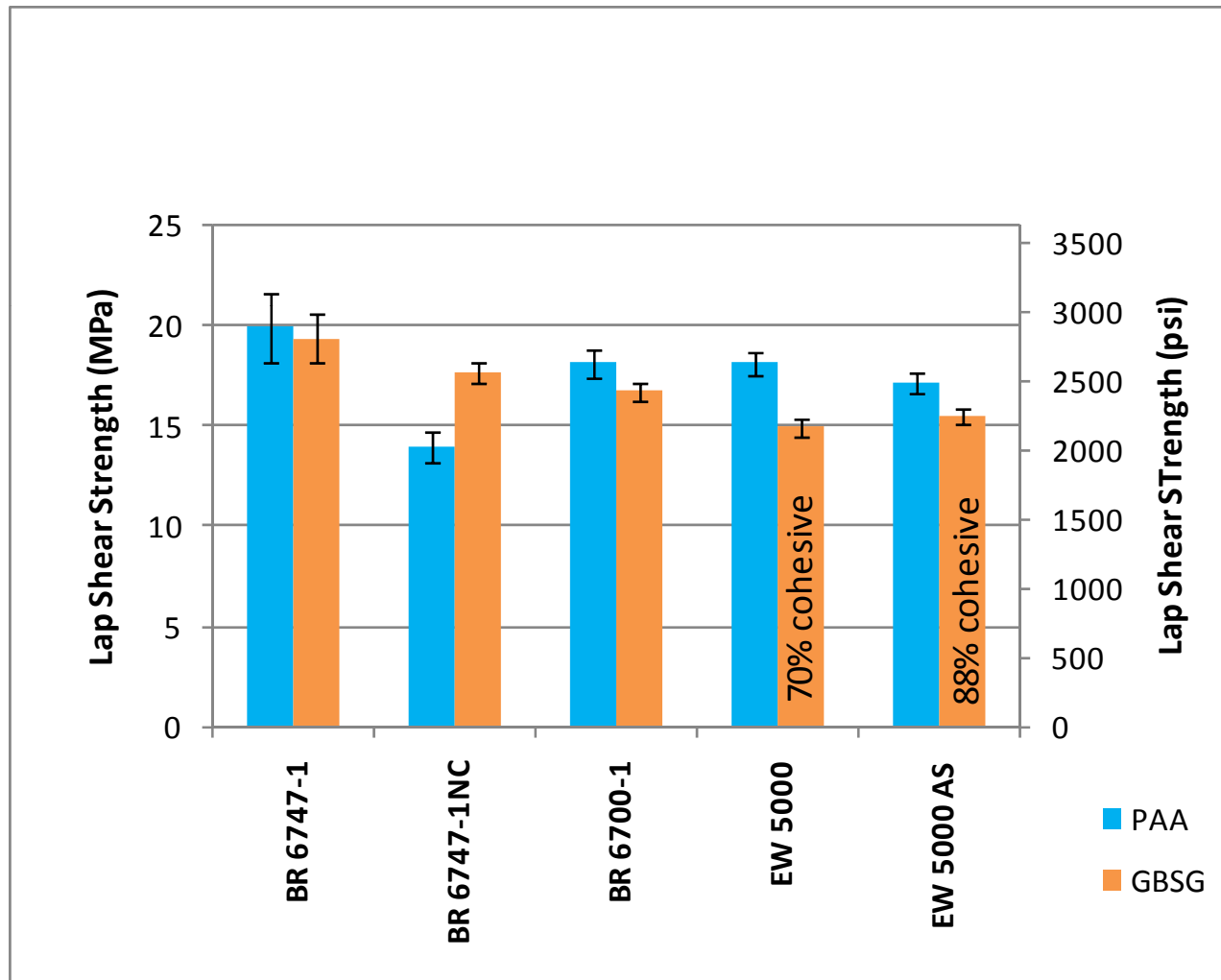
AF 500M Adhesive 22°C (72°F) Lap Shear





AF 500M Adhesive

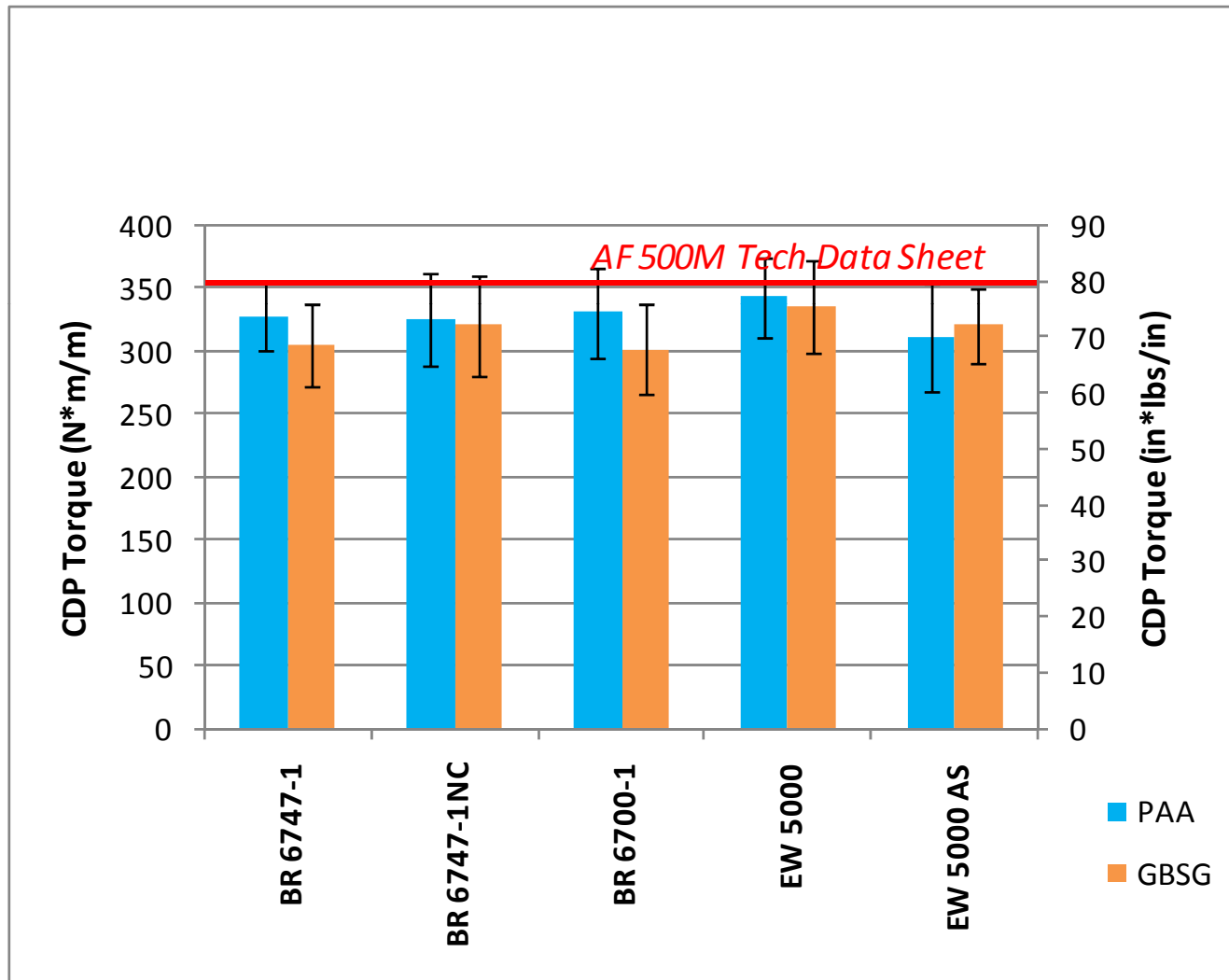
82°C (180°F) Wet Lap Shear





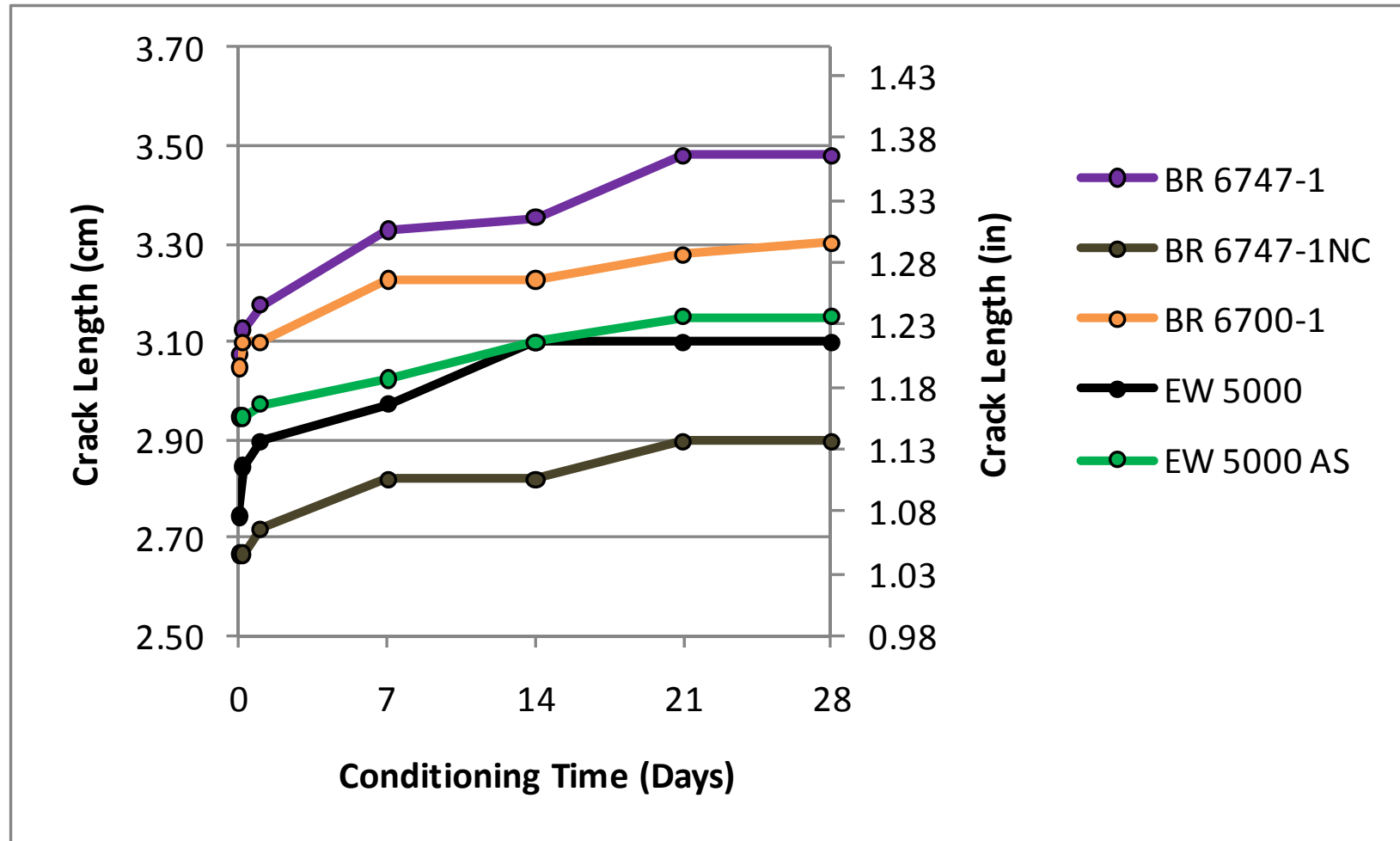
AF 500M Adhesive

22°C (72°F) Climbing Drum Peel





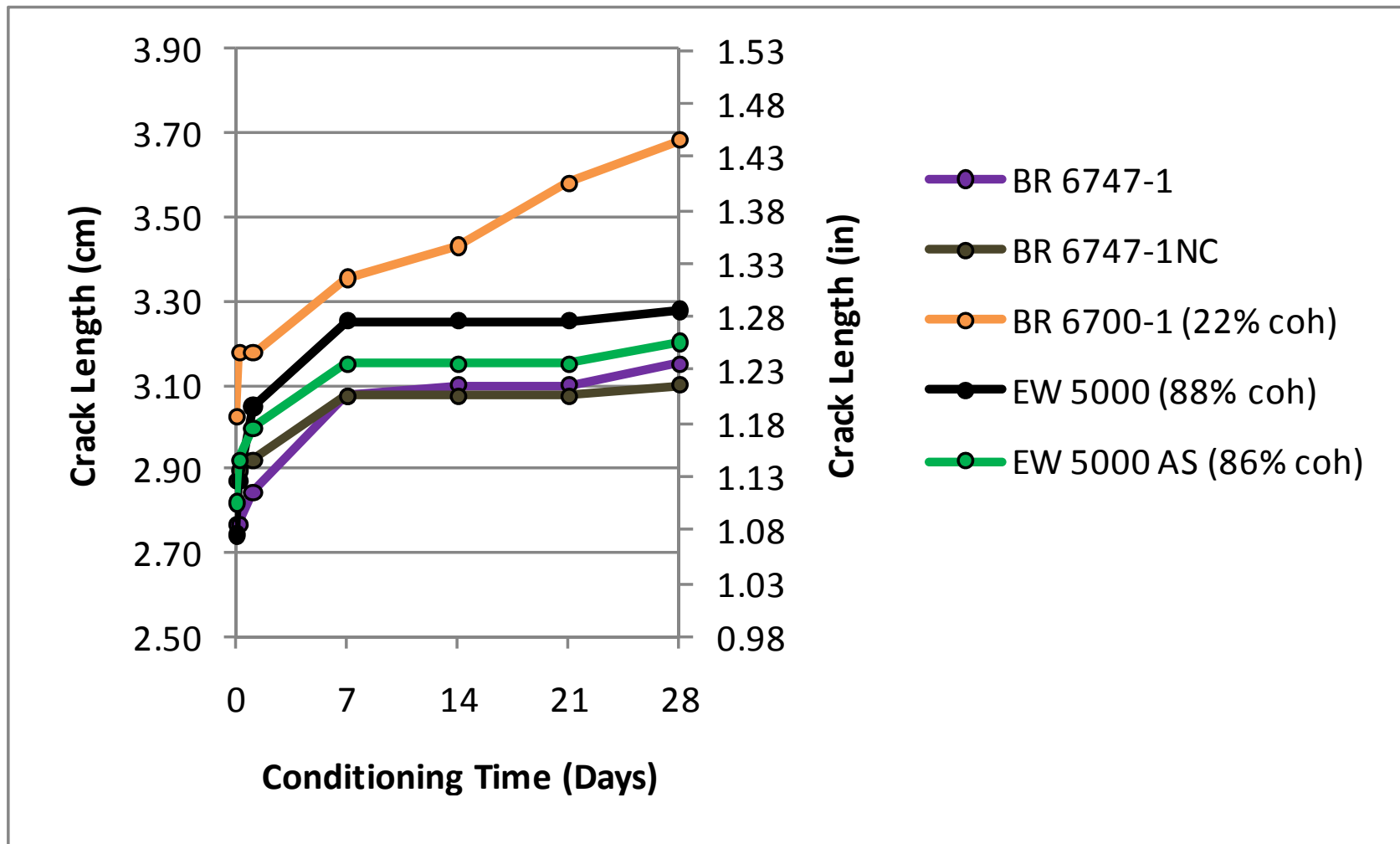
EA 9696 Adhesive with PAA 60°C (140°F) Wedge Test





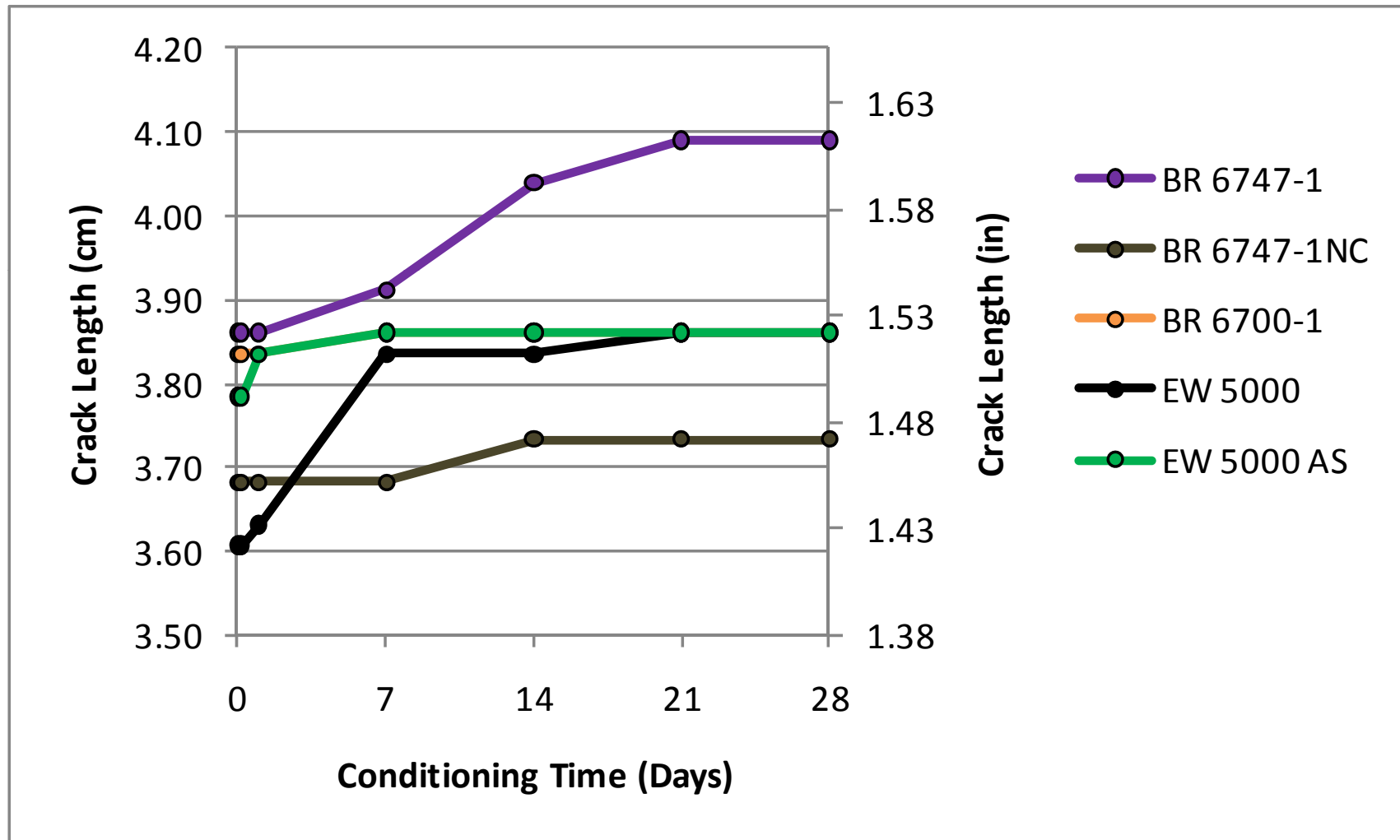
EA 9696 Adhesive with GBSG

60°C (140°F) Wedge Test



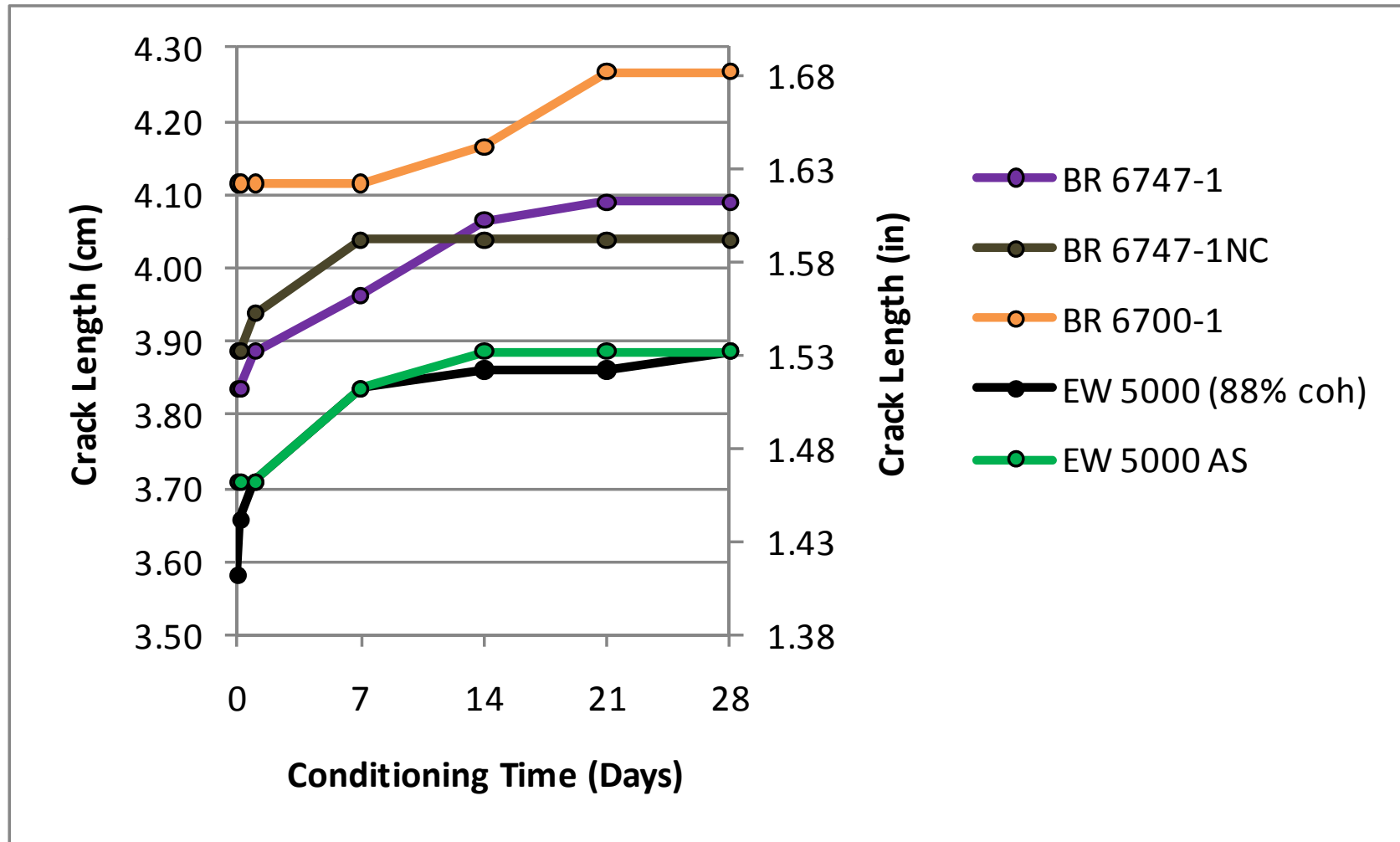


EA 9628 Adhesive with PAA 60°C (140°F) Wedge Test



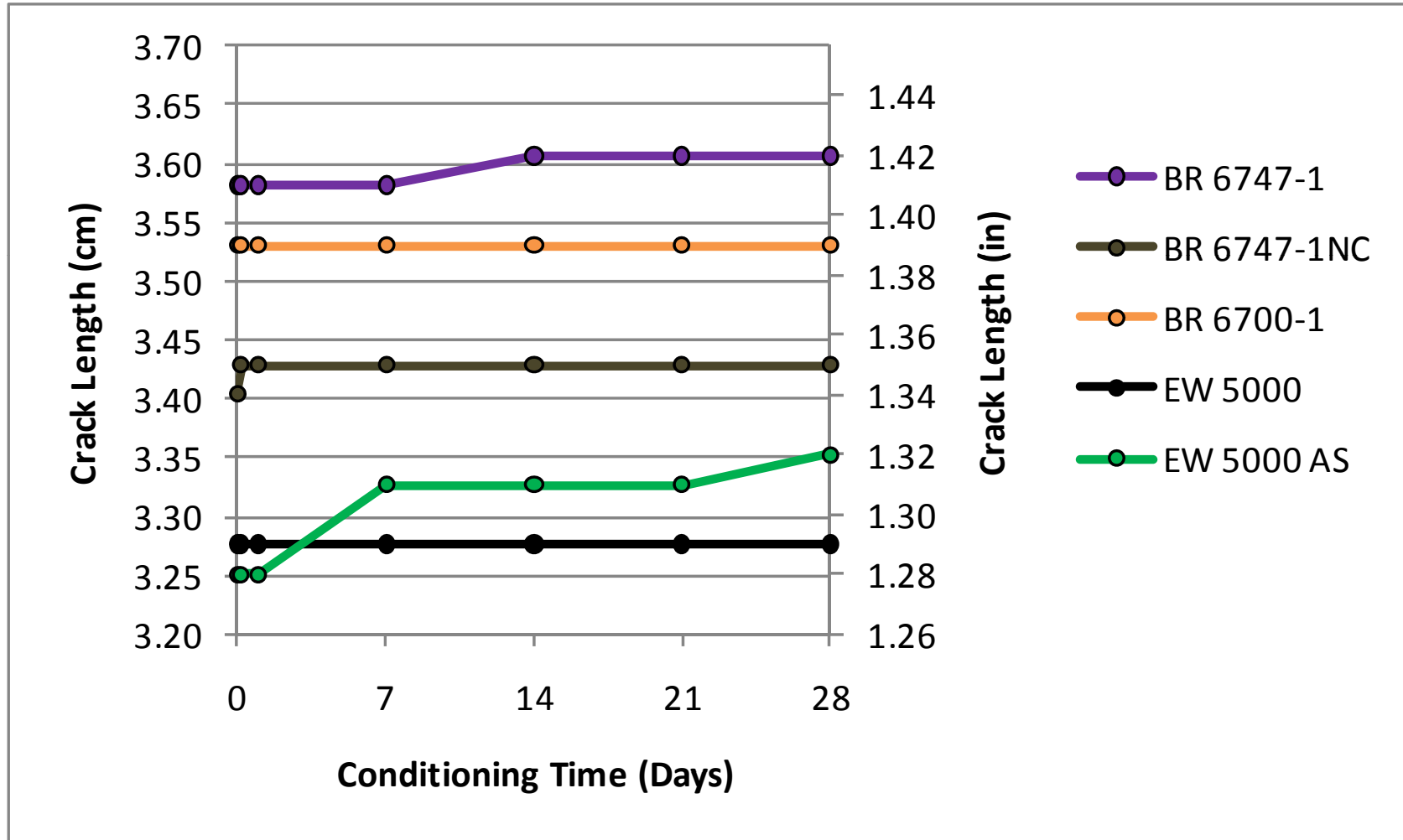


EA 9628 Adhesive with GBSG 60°C (140°F) Wedge Test





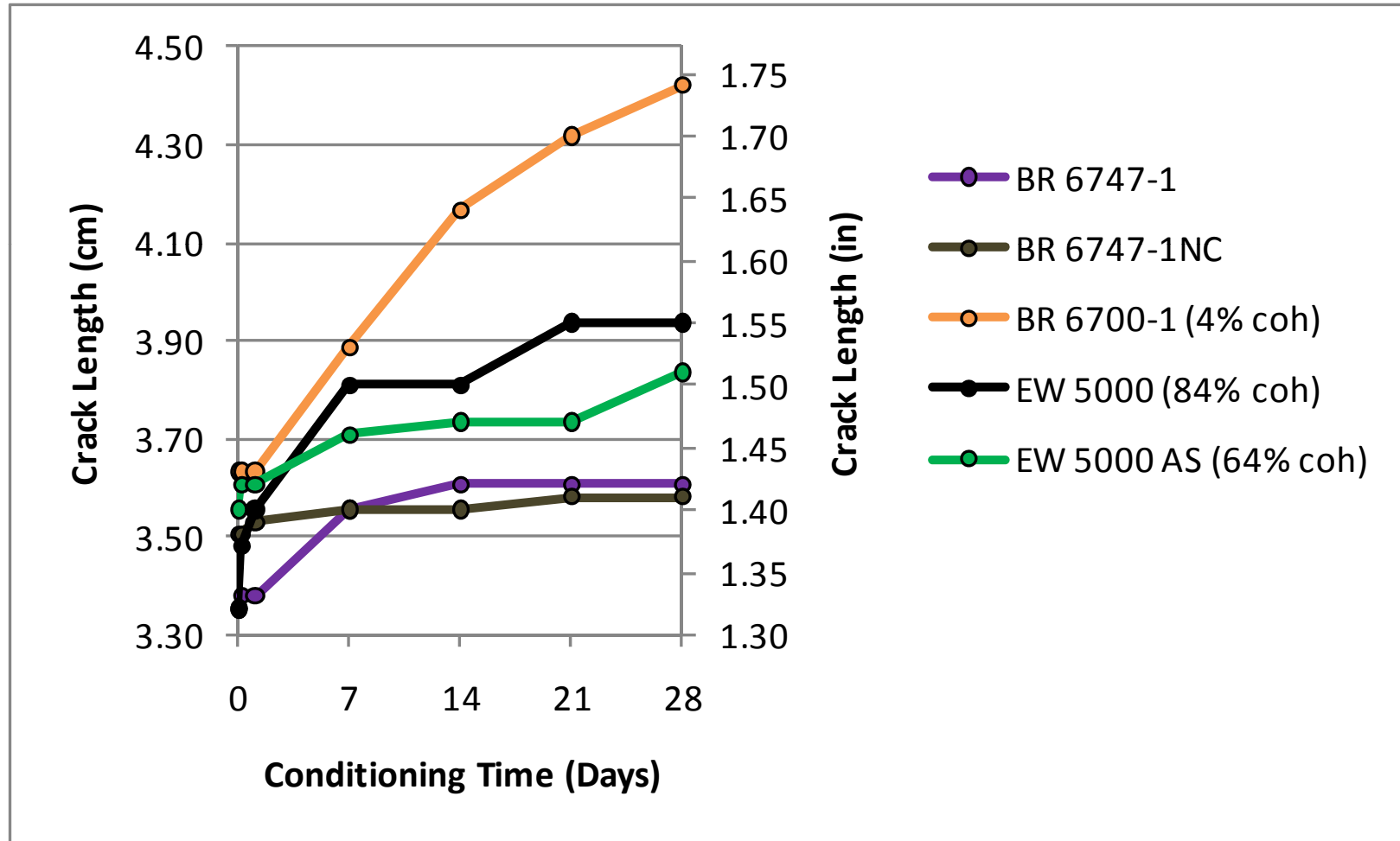
FM 73M Adhesive with PAA 60°C (140°F) Wedge Test





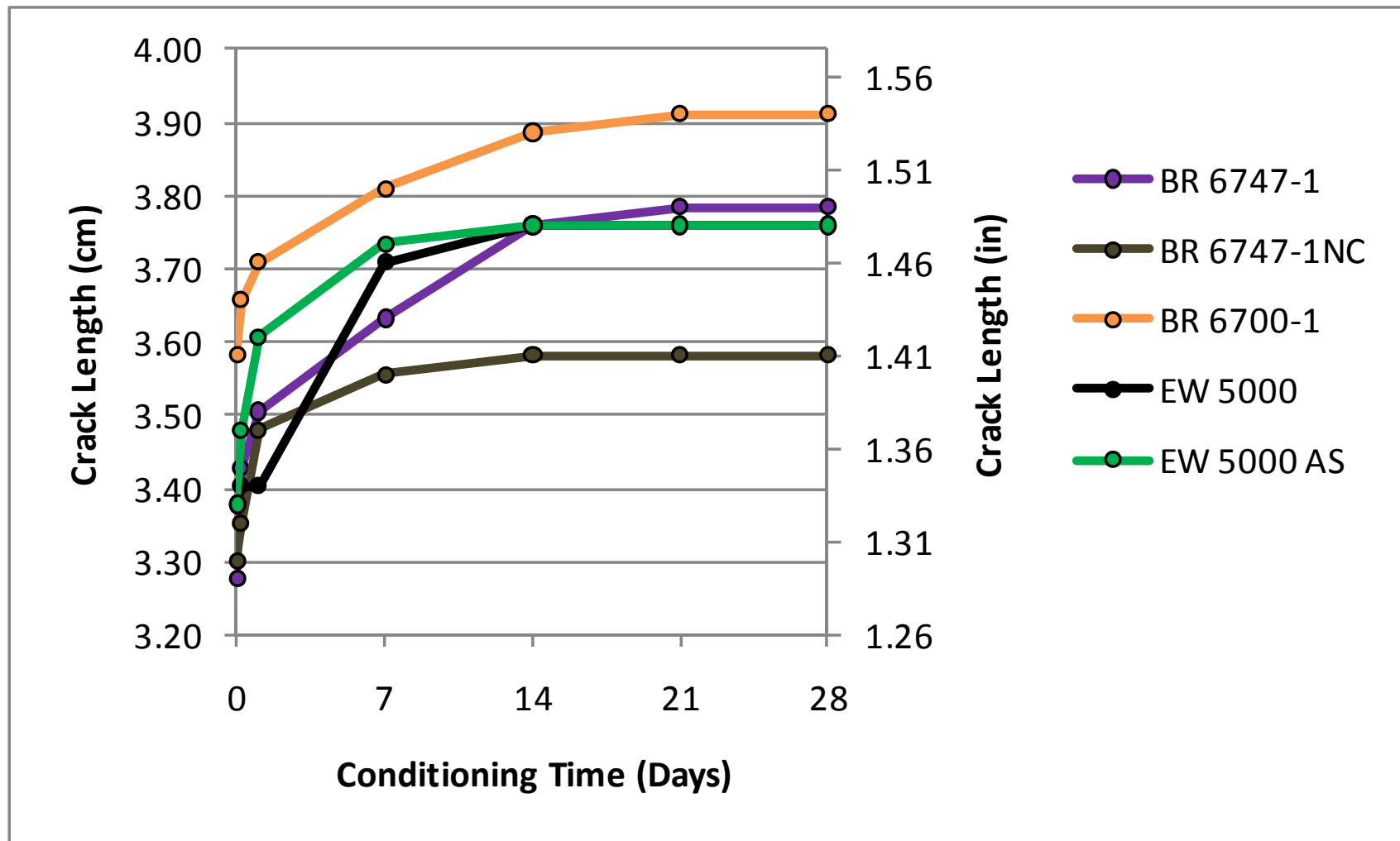
FM 73M Adhesive with GBSG

60°C (140°F) Wedge Test





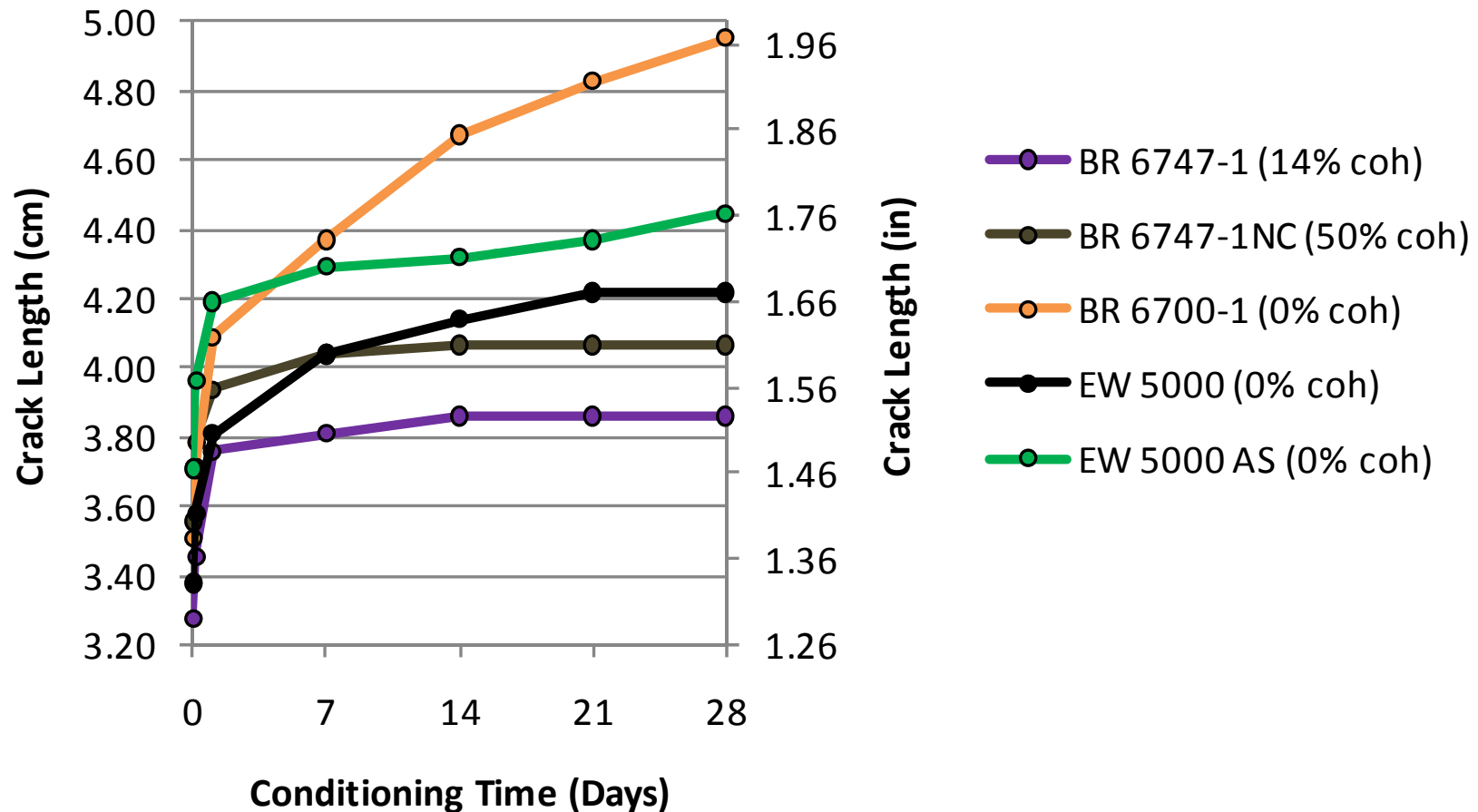
FM 209M Adhesive with PAA 60°C (140°F) Wedge Test





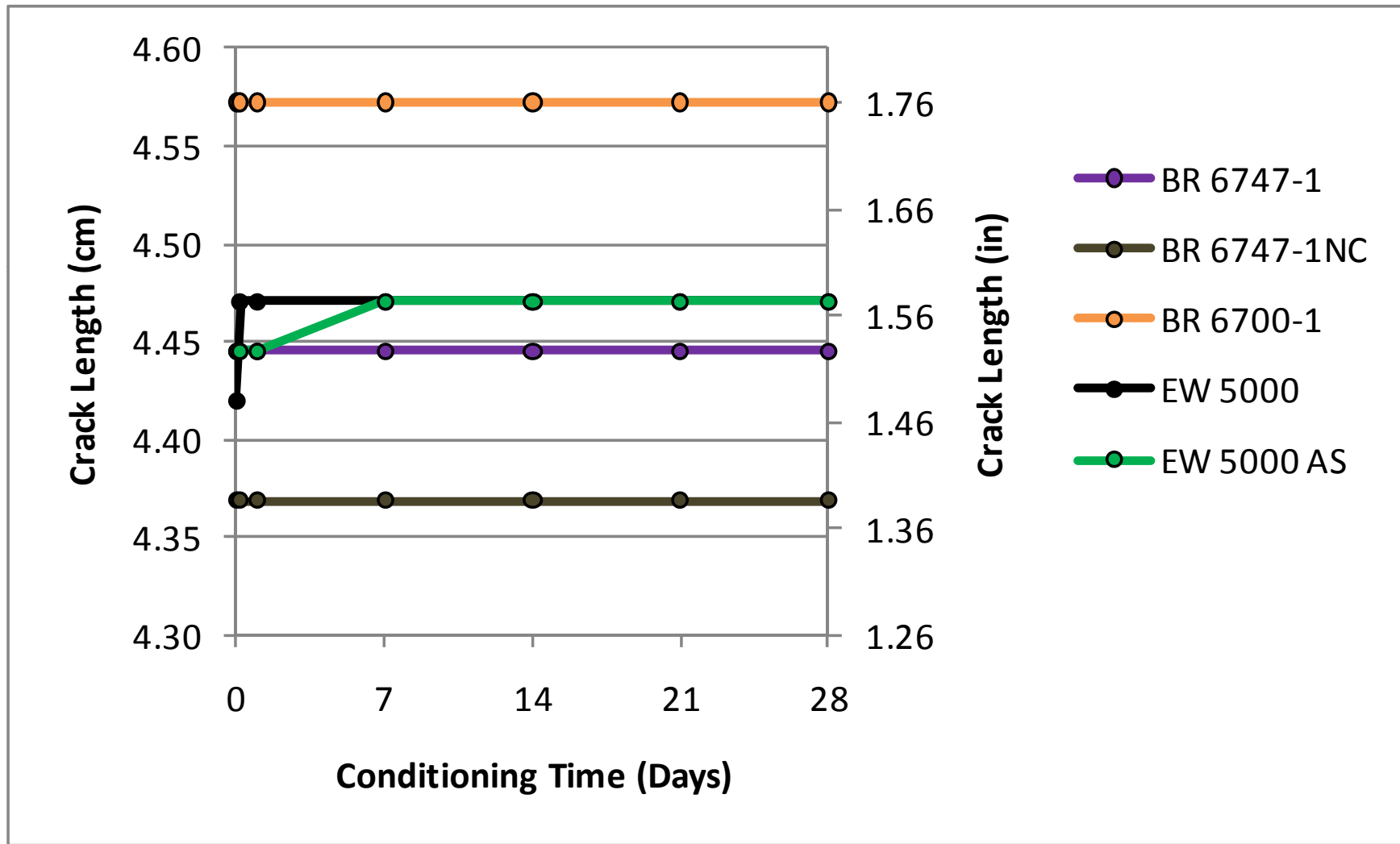
FM 209M Adhesive with GBSG

60°C (140°F) Wedge Test





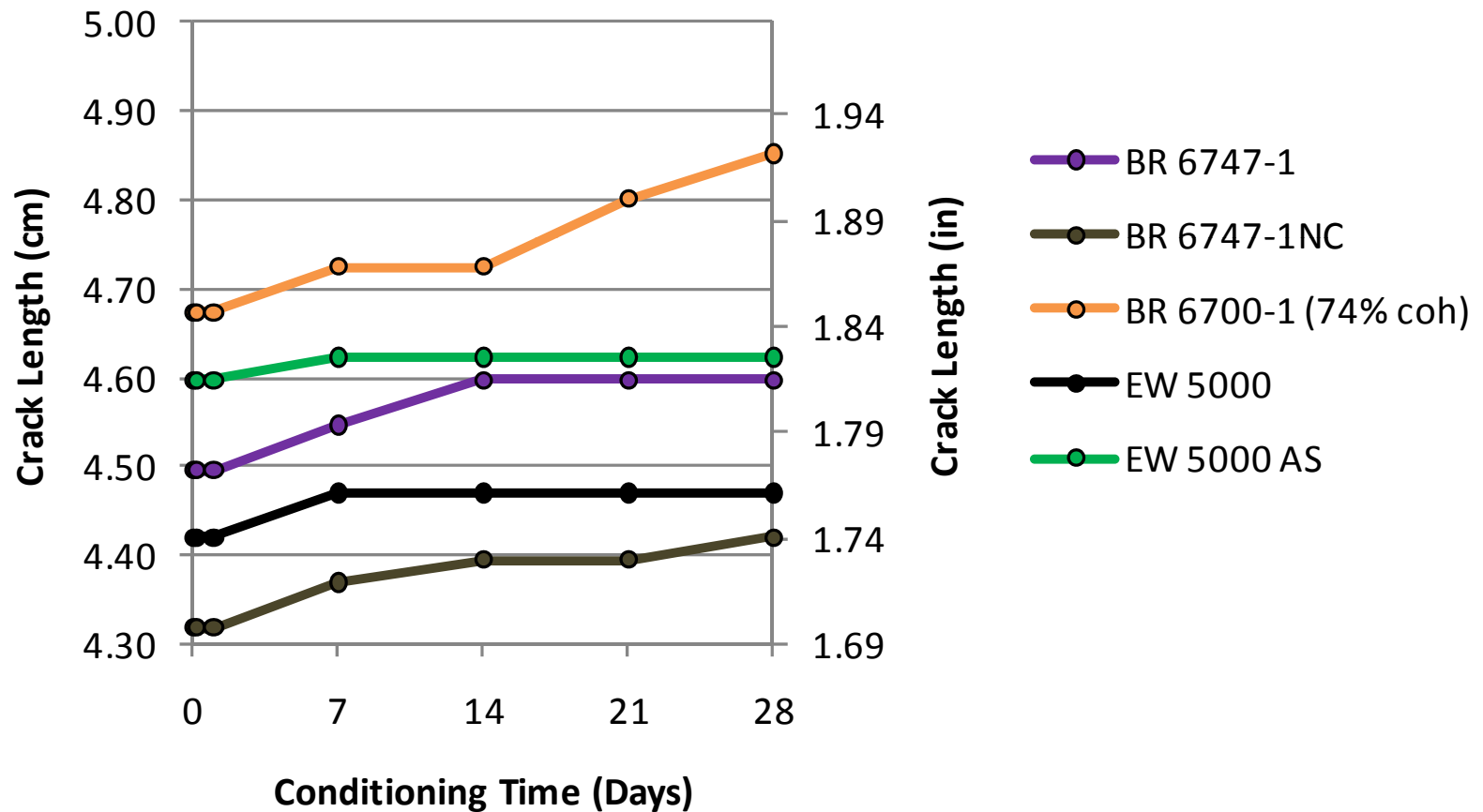
FM 300-2M Adhesive with PAA 60°C (140°F) Wedge Test





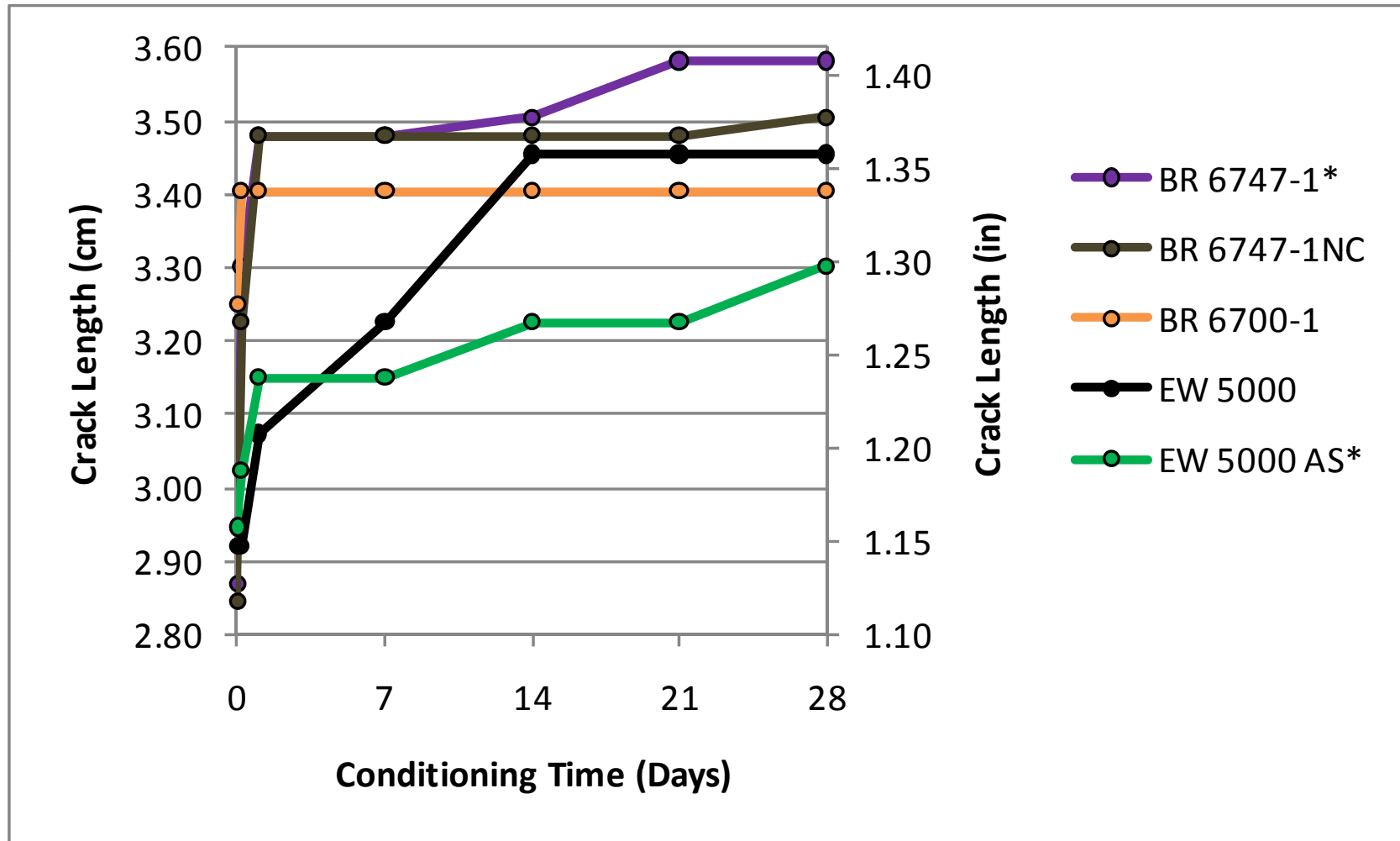
FM 300-2M Adhesive with GBSG

60°C (140°F) Wedge Test





AF 163-2M Adhesive with PAA 60°C (140°F) Wedge Test

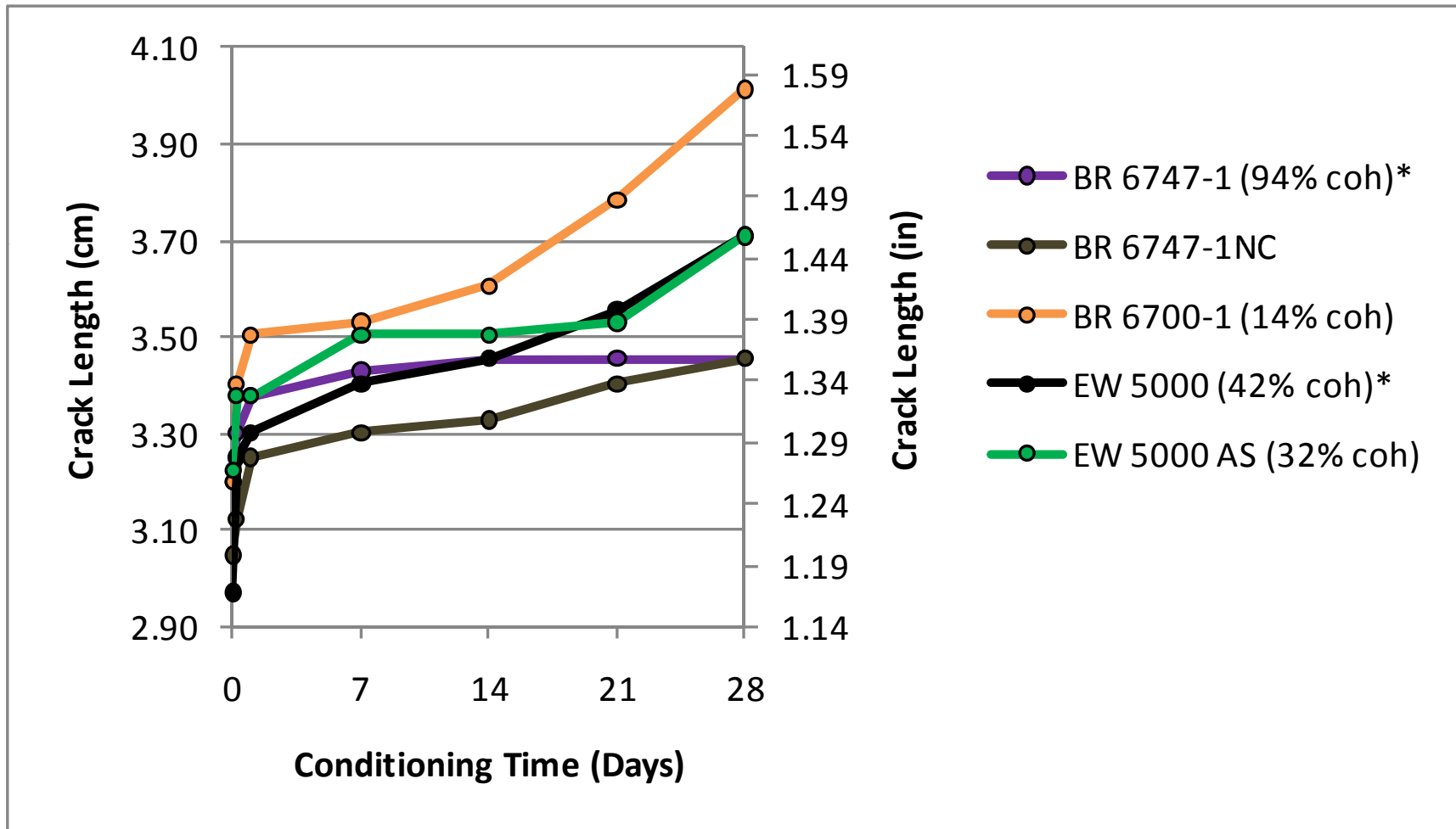


* Visually appeared to fail at the primer-adhesive interface, but a thin layer of AF 163-2M remains on the primer



AF 163-2M Adhesive with GBSG

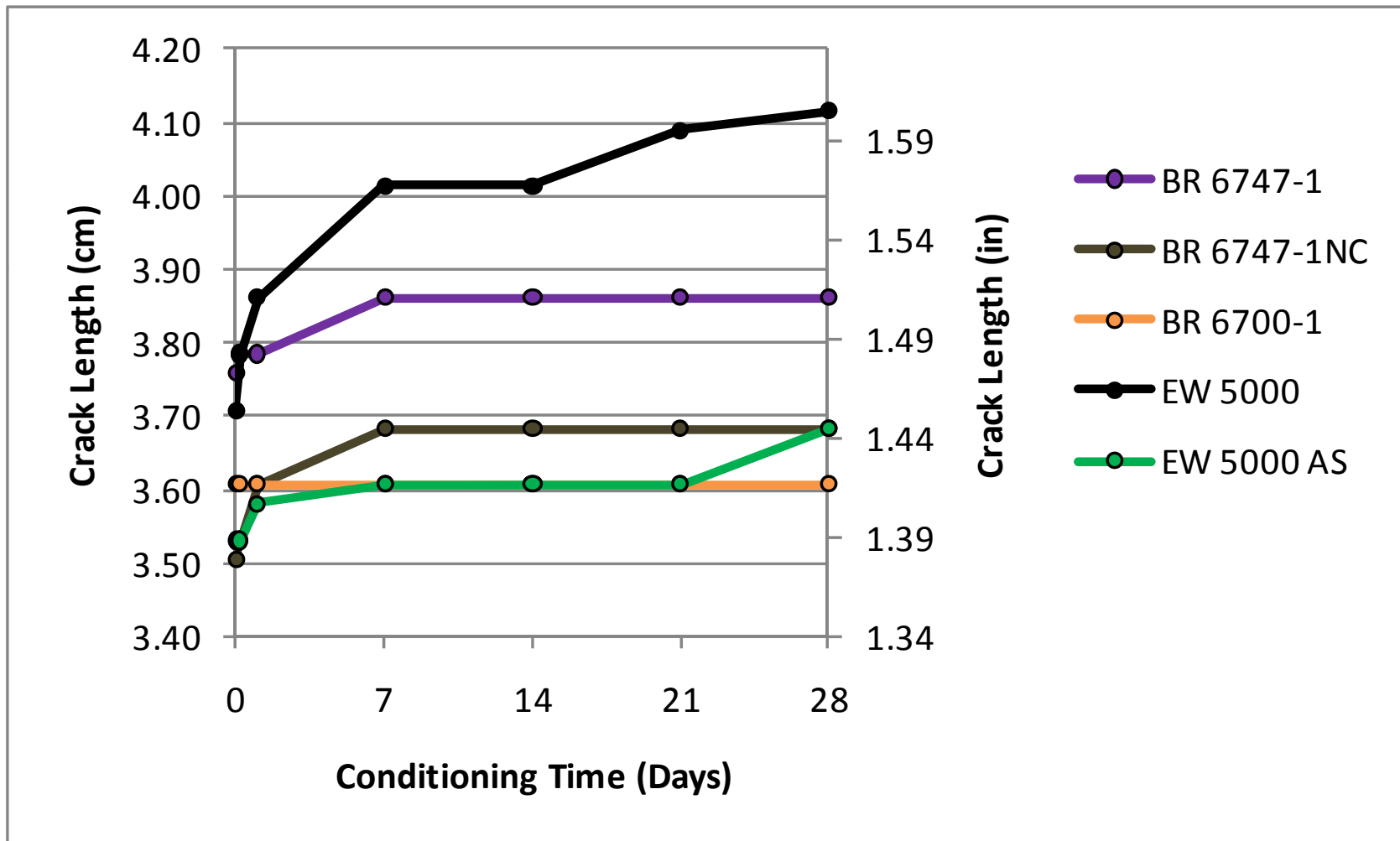
60°C (140°F) Wedge Test



* Visually appeared to fail at the primer-adhesive interface, but a thin layer of AF 163-2M remains on the primer



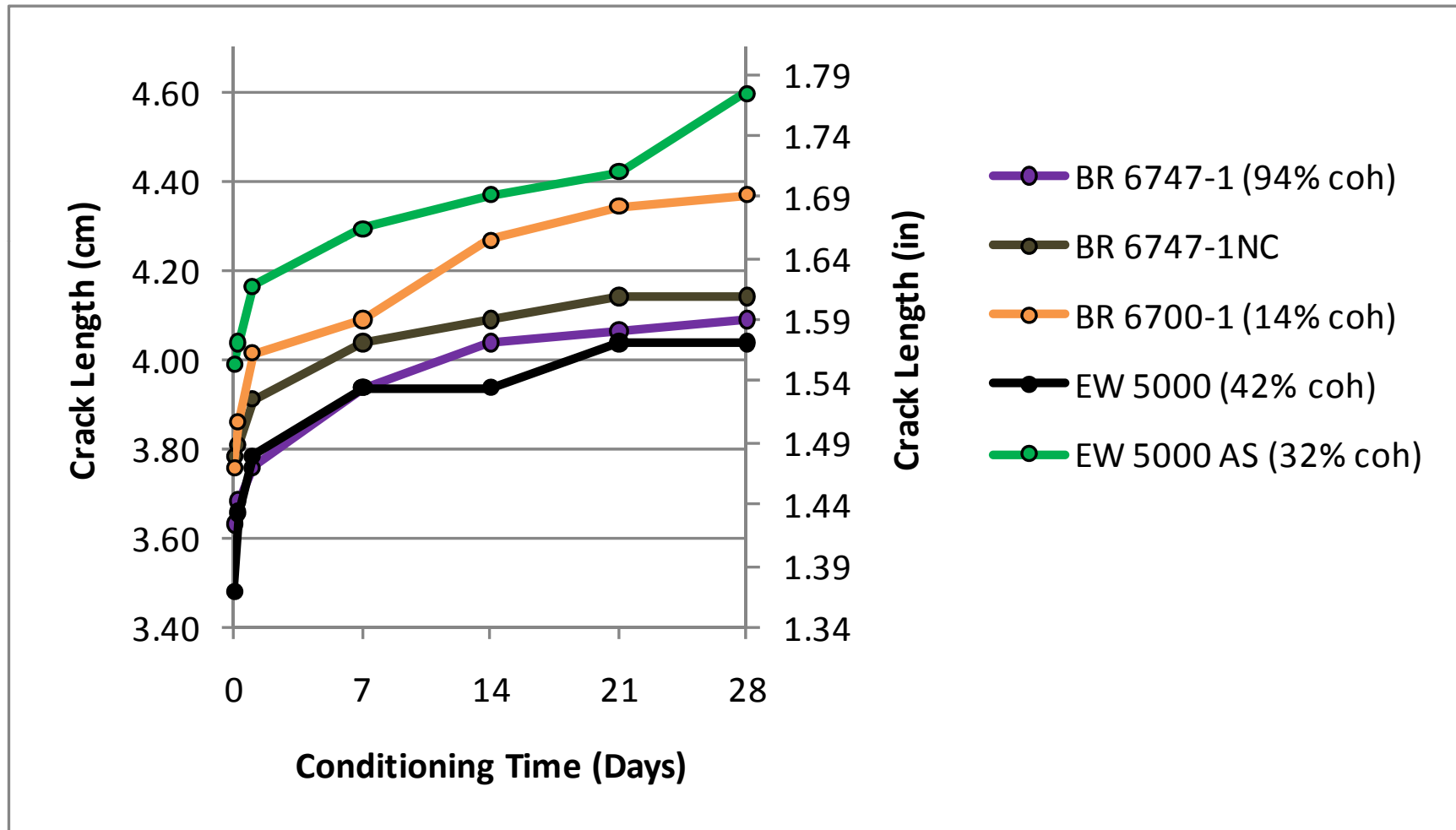
AF 500M Adhesive with PAA 60°C (140°F) Wedge Test





AF 500M Adhesive with GBSG

60°C (140°F) Wedge Test

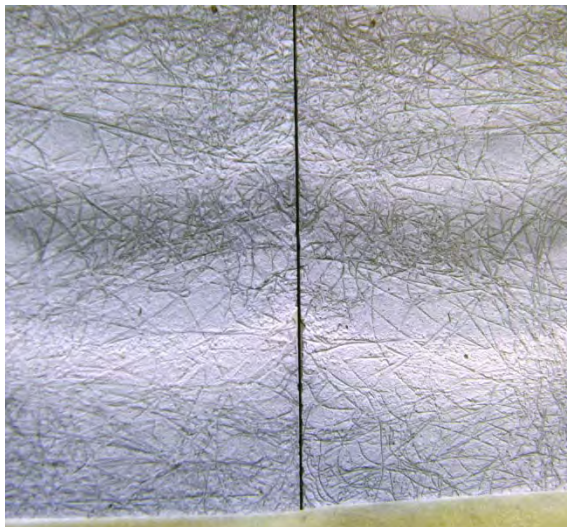




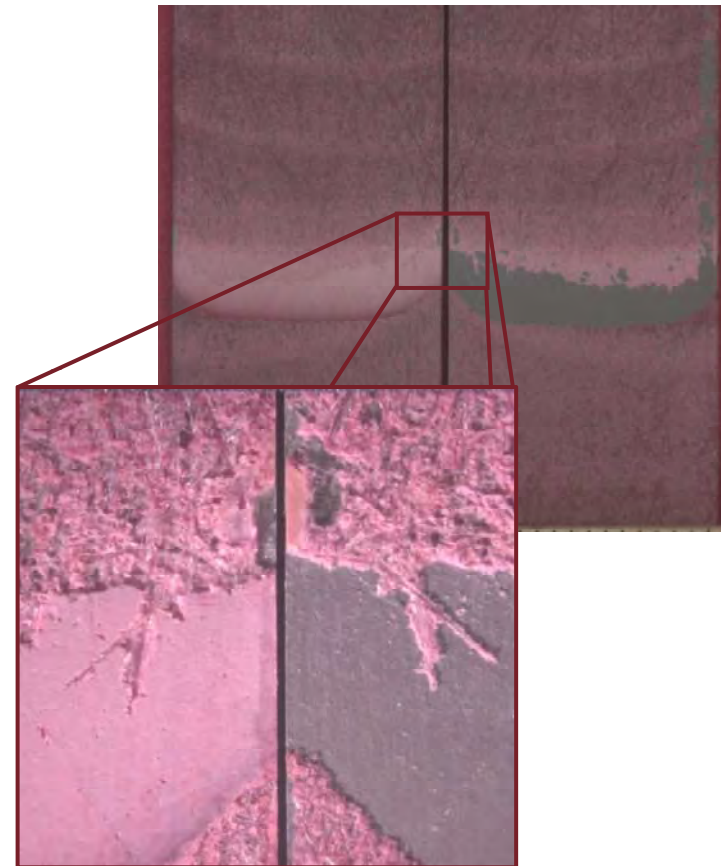
Typical Failure Modes Observed in 60°C (140°F) Wedge Tests



**Typical Primer-Aluminum Interfacial Failure
with FM 209M Adhesive**



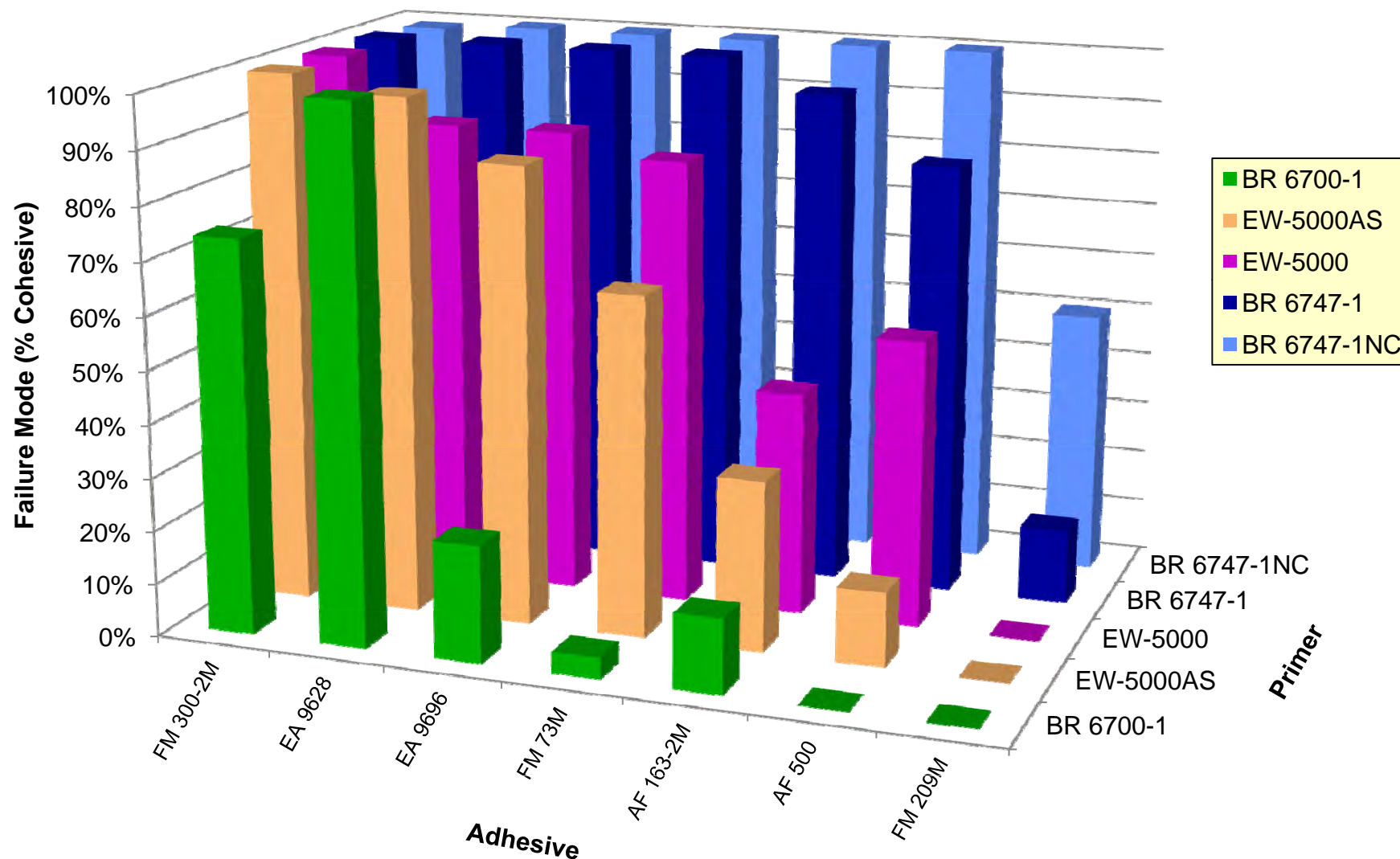
Typical Good "100 Percent" Cohesive Failure



**Typical AF 163-2M "100 Percent Cohesive Failure"
Near Primer-Adhesive Interface**



Summary of Failure Modes with GBSG in 60°C (140°F) Wedge Tests





Conclusions



- **Large Amount of Data Generated to Guide Future Efforts**
- **No Clear Path Forward for Non-Cr Primer Qualification**
 - Based on initial screening with only 5 specimens per test
- **PAA Test Results were Overall Superior to GBSG**
 - PAA is the premier aluminum surface preparation
 - PAA can be used with more combinations of surface preps & primers
- **Hot/Wet Lap Shear Results when Using GBSG Exhibit Poor Failure Modes**
 - Routinely seen even with good wedge tests data for same combination
 - A bit concerning
- **Wedge Test Results Must be Used with Caution**
 - Screening w/ controlled variables; no quantitative correlation to service



Conclusions (cont.)



- **BR 6747-1NC (No Corrosion Inhibitor) Yielded Amazingly Good Results**
- **BR 6700-1 Wedge Test Results with GBSG were Surprisingly Poor and Not Consistent with Previous AFRL (Good) Data**
- **FM 209M Wedge Test Data were Poor when with GBSG Prep**
- **FM 73M Generated Surprisingly Good Wedge Test Results with GBSG/BR 6747-1; Contradicts Previous USAF Data**
- **FM 300-2M Adhesive Generated Larger Initial Cracks in the Wedge Test and Tended to Produce Less Crack Growth**
 - Wedge test interrogates interface less stringently using FM 300-2M
- **AF 163-2M Adhesive Exhibits Failure Modes At or Near the Primer-Adhesive Interface with BR 6747-1 & Others**



Future Efforts



- **USAF Will Work with Industry-Government Non-Cr Adhesive Bond Primer Team to Further Evaluate Some of the Products Screened, as well as Promising New Candidates**
- **Another Industry-Government Team was Awarded a SERDP Project Aimed at Understanding the Requirements for Bond Primers and their Corrosion Inhibitors**
 - Investigate failed bonds and relationship to primer
 - Evaluate various types/levels of corrosion inhibitor in bond primers
 - Develop a risk assessment tool for bonded joints
 - Investigate alternative accelerated aging protocols
- **Much Work is Required to Implement any New Primer**
 - Laboratory testing beyond screening
 - Generation of data based on field-level processing